

## **ATTACHMENT 3**

### **The CAMDS Site Laboratory and Monitoring Quality Control Plan**

**November 25, 2008**

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## **1.0 PURPOSE AND GOALS**

The purpose of this document is to provide direction for all Quality Control (QC) activities related to the monitoring and analytical functions performed at the Chemical Agent Munitions Disposal Systems (CAMDS). The U.S. Army Chemical Materials Agency (CMA) developed a programmatic Laboratory and Monitoring Quality Assurance Plan (LMQAP) and the Monitoring Concept Plan (MCP) to define the minimum requirements for laboratories and monitoring teams to implement a quality system to support CMA activities. This site specific CAMDS Laboratory Monitoring Quality Control Plan (CLMQCP) describes the quality control program to be implemented by the Analytical and Monitoring Divisions of the CAMDS Site.

Any discrepancies or deviations from the requirements set forth in this Attachment shall be submitted to the Chief, Monitoring Division and the Director of Operations Support for review. Approval by the Executive Secretary is required before any modification of this Permit (R315-3-4).

### **1.1 Scope**

This document outlines the purpose, policy, organization, and operations of all quality assurance/quality control (QA/QC) programs that have been established to support the CAMDS Site.

The CAMDS Site Laboratory and Monitoring Quality Control Plan (CLMQCP) addresses the monitoring and laboratory analytical requirements for; 1) the processing of agent contaminated secondary waste, 2) the MDC-2 A&B ovens, 3) the Deseret Chemical Depot (DCD) Perimeter monitoring program, for agent stored and processed at DCD, and 4) Mustard, GA and Lewisite monitoring and analytical support for TOCDF CAL laboratory.

The requirements of this CLMQCP are applicable to all government and contractor and subcontractor personnel performing agent and non-agent monitoring, analyses, and related quality control activities at the CAMDS Site.

Specifically, the CLMQP shall establish procedures for inspections, agent monitoring systems, recordkeeping, equipment maintenance, sample collection, laboratory analytical methods, personnel training, and Quality Control/Quality Assurance. The CAMDS Site shall meet or exceed all Federal, State, and Local regulatory requirements for the safe, secure, environmentally correct storage, handling, and destruction of secondary waste entrusted to the CAMDS Sites care. The CAMDS Site shall continually improve the effectiveness of their quality management system and initiate, recommend, and provide quality solutions for improvements. Established procedures shall ensure:

1. A consistent framework for the generation of analytical data to activities conducted by the CAMDS Site.
2. Demonstrate that analytical systems are in control.
3. The quality of each analytical system including precision, accuracy, and sensitivity is sufficient for the needs of each project.
4. Deficiencies in data quality shall be identified and corrected in a timely manner.

5. Enable the user to identify and implement actions that are necessary to ensure the validity of data.
6. Providing accurate records, which document sample collection, equipment maintenance, analytical methods and implemented Quality Control measures.
7. That the Proficiency Testing Program (PTP) shall provide an objective means of assessing and determining the reliability of the data produced.

## **1.2 Management Review**

DCD Risk management shall review the organization's quality management systems (QMS), annually. The Operational support management shall review Quality systems monthly, to ensure its continuing suitability, adequacy, and effectiveness. Records of all reviews shall be kept at the appropriate office.

## **2.0 ORGANIZATIONAL DESCRIPTION**

The Operations Support Directorate organization is responsible for ensuring that all quality guidance is implemented and followed.

The Directorate consists of the Laboratory Analytical Division and the Monitoring Division. These Divisions support the CAMDS Site, selected monitoring stations in Area 10, TOCDF CAL laboratory, and DCD perimeter monitoring.

Appendix A provides detailed descriptions of personnel responsibilities for the Operations Support Directorate.

## **2.1 The Laboratory Analytical Division**

The major duties that are performed by the CAMDS Site laboratory are:

1. Depot Area Air Monitoring System (DAAMS) tube analysis for agents, methylethylphosphonofluoridate (GB), Ethyl N, N-dimethylphosphoramidate-cyanidate (GA), O-ethyl S-(2-diisopropylaminoethyl) methylphosphonothiolate (VX), Levisite Mustard, bis-(2-chloroethyl) sulfide (HD), and dichloro-2-chlorovinyl arsine (Lewisite).
2. Process sample analysis, standards preparation, and Agent accountability.
3. Maintain RDTE Agent Lab, log of the reciprocal of the hydrogen ion concentration (pH), flashpoint, available chlorine, percent (%) carbonate, % Bicarbonate, % sodium hydroxide, total solids, total suspended solids, total dissolved solids, trinitrotoluene (TNT), cyclotrimethylenetrinitramine (RDX), a class A explosive, trinitrophenylmethylnitramine (TETRYL), nitroglycerine, chemical agent concentrations, and chemical agent impurities.

## **2.2. The Monitoring Division**

The Monitoring Division provides the monitoring support for the CAMDS Site, DCD Perimeter, and Area 10 and TOCDF CAL Laboratory. Before each individual agent campaign the monitoring system is operated in the configuration in which it will be used during the campaign. Agents monitored for are VX, GB, GA, Lewisite and Mustard. The major duties that are performed are: DAAMS operations/collection, operation/maintenance of Near Real Time (NRT) monitors, Hewlett Packard Dynatherm Systems (HPD) for use in stationary and mobile

configurations, Real Time Analytical Platforms (RTAP)s, and monitoring equipment/station maintenance.

### **3.0 LABORATORY & MONITORING INSTRUMENTATION**

#### **3.1 Gas Chromatograph (GC)**

Samples are placed into a heated injection port and are rapidly heated and vaporized. A stream of gas (Helium) carries each sample along a column that contains a stationary phase. The sample becomes distributed between the mobile gas phase and the stationary phase. Different components of the sample are separated in the column. The higher a substance's affinity, in a sample, for the stationary phase, the more slowly it goes through the column. Different column phases will change the elution order of the compounds.

The GCs in the laboratory are equipped with either a DAAMS inlet or a standard liquid injection port. The GCs are equipped with a Mass Selective Detector (MSD), Flame Photometric Detector (FPD), and Flame Ionization Detector (FID).

##### **3.1.1 Mass Selective Detector (MSD)**

The MSD is a universal detector that is capable of detecting hundreds of different compounds in a sample. The sample enters the MSD after the sample is eluted through the GC column. Each compound of the sample enters the ionization chamber and compounds are fragment into several ions. The ions are then sorted according to their mass by the quadrapole. At the end of the quadrapole, the ions hit a detector and an amplifier connected to the detector, which produces an electronic signal. The resulting signals are recorded by a computer and when graphed, create a mass spectrum.

The sample spectra can be compared with a spectral library of known chemicals and the individual compounds in a sample may be identified.

The MSD is used to identify interferents and also used to confirm the presence of chemical agent.

##### **3.1.2 Flame Photometric Device (FPD)**

All routine DAAMS analyses are performed using a GC equipped with an FPD. The FPD is used to measure compounds containing phosphorous and sulfur. The sample is burned in a hydrogen/air flame in the FPD, after elution from the GC column. Phosphorous and sulfur compounds emit a specific wavelength of light when burned. This light will then pass through a band pass filter, which is designed to only transmit specific wavelengths. The light that is transmitted is passed into the photo multiplier and amplified to produce an electronic signal. The amount of signal is proportional to the concentration of the analyte.

##### **3.1.3 Flame Ionization Detector (FID)**

The FID is used to measure organic compounds. The sample is mixed with hydrogen and then routed through a stainless steel jet in the FID, after elution from the GC column. The sample is burned in a flame, which ionizes the compounds. A collector electrode attracts the negative ions to the electrometer amplifier producing an analog signal, which is connected to a data collection software system.

The laboratory uses an FID to perform agent standard degradation monitoring. The FID is a universal detector because the flame will ionize most compounds and produce a signal.

#### **3.1.4 High Performance Liquid Chromatography Mass Spectrometry (HPLC-MS)**

A sample is introduced into the HPLC-MS and is carried via a high-pressure liquid through a column that contains a stationary phase. The sample becomes distributed between the mobile liquid phase and the stationary phase. The higher a substance's affinity for the stationary phase, the more slowly it will exit the LC column. The sample compounds then enter a nebulizer and are sprayed into a chamber where the compound becomes ionized. The ionized molecules are either negatively or a positively charged. The ions are then separated into different masses in the quadrapole. These charged ions hit a detector, at the end of the quadrapole; their signal is amplified and recorded as an electronic signal. A mass spectrum is then graphed from this data. Any vapor validation studies must be approved by the Executive Secretary before use.

#### **3.1.5 Inductively Coupled Plasma Mass Spectrometry (ICP/MS)**

Unlike a GC or an LC, the ICP/MS does not separate the components of a sample prior to the sample hitting the detector. Currently, this instrument is not in use at the CAMDS Site. A completed validation study must be approved by the Executive Secretary before use.

### **3.2 Monitoring Instrumentation**

#### **3.2.1 Near Real Time Monitors (ACAMS/MINICAMS)**

The Near Real Time (NRT) Monitors are configured to monitor one or more of the chemical agents being processed at the Site. NRT monitors are used to monitor the operational areas, filters, and the MPF stack. NRT ACAMS and MINICAMS monitor for chemical agents GA, GB, VX, Mustard, and Lewisite at the STEL, SEL, and IDLH concentrations levels.

The NRT monitors are an automated gas chromatograph (GC) that first collects agent on a solid sorbent and then thermally desorbs the agent into a separation column for analysis. The components eluting from the column are detected by a flame-photometric detector (FPD) or a halogenated selective detector (XSD), which can respond to compounds containing either phosphorus [e.g., Isopropylmethylphosphonofluoridate (GB) and VX or sulfur (Mustard)].

Because of the low volatility of VX, two conversion pads must be used for the detection of VX where the sample enters the monitoring system. The pad converts VX to a more volatile compound (the G-analog of VX, ethyl methylphosphonofluoridate), which can be quantitatively transferred through the sampling and analysis system.

#### **3.2.2 Depot Area Air Monitoring System**

Depot Area Air Monitoring System (DAAMS) collects air samples to detect chemical agents GA, GB, VX, Mustard, and Lewisite for confirmation of NRT alarms and as primary historical monitors in areas not monitored by NRT monitors. Analysis of DAAMS samples provides confirmation of agent if concentrations are at or above the defined Reporting Limit (RL). Instrumentation used for agent confirmations are the Gas Chromatography/Flame Photometric Detector (GC/FPD) or GC-Mass Selective Detector (GC-MSD).

### **3.2.3 Real Time Analytical Platform (RTAPS)**

Real Time Analytical Platforms are mobile NRT stations used in situations where NRT monitoring is required but the location does not have any temporary (fixed) or permanent stations.

## **4.0 MONITORING CONFIGURATIONS AND AREAS MONITORED**

Agent monitoring systems are used and configured in conjunction with a variety of engineering systems to provide process control and interlocks in applicable agent storage and worksite activities. The objectives of agent monitoring are to ensure the protection of the workers and the environment. Tables 4-1, 4-2, 4-3, and 4-4, provide rapid monitoring systems response times and historical and perimeter sampling times for AEL levels.

### **4.1 Monitoring Configuration**

There are three monitoring configurations employed at the CAMDS Site; 1) NRT stations only, 2) NRT co-located with Depot Area Air Monitoring System(s) (DAAMS) and, 3) DAAMS only stations. A brief description follows:

#### **4.1.1 NRT Only Stations**

NRT only stations are primarily used in toxic areas to provide information to management for protective clothing determination for entries into toxic areas.

#### **4.1.2 NRT Monitors and Co-located DAAMS Stations**

NRT monitors with co-located DAAMS stations have dual purposes as follows:

1. The NRT monitors are the primary monitors, which provide an early warning system to plant personnel of potential agent release above the alarm set point. The co-located DAAMS are used to either deny or confirm the presence of agent during the NRT alarm period.
2. The DAAMS tubes are also analyzed at the WPL (12 hour), regardless of an NRT alarm to identify any low-level agent contamination. The MPF stack is monitored with NRT monitors at the SEL and at a frequency of every four hours with DAAMS. The Filter Stacks are monitored with NRT monitors at the STEL and with DAAMS at the WPL every 12 hours.

#### **4.1.3 DAAMS Station Only**

Historical DAAMS stations are used in areas not expecting to have agent and are monitored at the WPL and the GPL. The DAAMS historical tubes are not connected to an alarm.

### **4.2 CAMDS Monitoring Areas**

In the demilitarization plant area, hazard category classifications and personnel occupancy are the factors used to determine monitoring activities. When monitoring is conducted for personnel protection or to assess potential personnel exposure, the monitoring must be sufficient to identify, verify, and quantify the agent. STEL and WPL monitoring is performed in areas of the facility where workers may have a potential exposure to chemical warfare agent.

#### 4.2.1 CWM and Hazardous Waste Process Areas and Airlocks (Hazard Category A and B)

Toxic and Process areas are potentially contaminated with liquid agent or agent vapor. These areas are monitored using NRT monitors. No confirmation monitoring is necessary because the presence of chemical agent is expected. The agent concentrations determine the Personnel Protective Equipment (PPE) that is required for personnel entry. At times, the monitoring may be enhanced to allow the PPE for specific toxic areas to be reduced to enhance worker mobility. Reduction of PPE requires Safety Officer approval. Enhanced monitoring may include changing the NRT monitors to a more sensitive detection level or by adding DAAMS in order to confirm or deny an NRT alarm.

Toxic and Process area airlocks serve as access/egress points between contaminated areas (agent or other hazardous wastes) and clean work areas. To limit the transfer of hazardous waste from "toxic" areas to "work areas", under normal conditions, items and personnel are cleared through an airlock or egress area. Adherence to written procedures governing the proper egress from areas where hazardous waste is being processed is required. These procedures shall ensure that equipment, clothing, or personnel exiting hazardous waste process areas do not spread hazardous waste outside of these areas. All personnel involved in toxic/process area operations shall be trained in proper egress procedures. Egress procedures shall include both proper NRT monitoring to confirm chemical agent readings, are less than the action level, where required, but also procedures to ensure all types of hazardous waste are not transferred outside the process areas.

#### 4.2.2 Process Support Areas (Hazard Category C)

The C areas are any rooms, mechanical rooms, and observation corridors that are connected directly via ventilation to hazard category A and B areas. These areas are monitored with NRTs at the STEL and at the WPL level daily.

Table 4-1: High-Level Agent Detector Response Times.		
AGENT	DETECTOR	RESPONSE TIME TO IDLH/GLD LEVELS (minutes)
GB	NRT 1	2.17 IDLH
VX	NRT 1	2.17 IDLH
H	NRT 1	2.17 GLD

Table 4-2: Low-Level Detector Rapid Response Times to STEL Levels.		
AGENT	DETECTOR	RESPONSE TIME to STEL LEVELS (minutes)
GB	ACAMS <sup>(1)</sup>	3
GB	MINICAMS™	5
VX	ACAMS	5
VX	MINICAMS™	3
H	ACAMS <sup>(1)</sup>	5
H	MINICAMS™	5
L	MINICAMS™	10
(1) SEL Monitoring Levels are the same response time.		

<b>Table 4-3: Low-Level Rapid Response Detectors to SEL Levels.</b>		
<b>AGENT</b>	<b>DETECTOR</b>	<b>RESPONSE TIME to SEL LEVELS (minutes)</b>
GB	ACAMS	3
GB	MINICAMS™	5
VX	ACAMS	5
VX	MINICAMS™	5
H	ACAMS	3
H	MINICAMS™	5

<b>Table 4-4: Sampling Times of Historical and Perimeter Monitors.</b>			
<b>AGENT</b>	<b>SAMPLING SYSTEM</b>	<b>AGENT EXPOSURE LIMIT</b>	<b>SAMPLING TIME (hours)<sup>(1)</sup></b>
GA/GB	DAAMS	WPL	2 or 12
		SEL	4
		GPL	24
VX	DAAMS	WPL	2 or 12
		SEL	4
		GPL	24
H, L	DAAMS	WPL	2 or 12
		SEL	4
		GPL	12

(1) 12-Hour samples are used to certify laundry and protective equipment are clear of agent and to certify Life Support System (LSS) air. This certification of LSS air is good for 24 hours.

#### **4.2.3 Non-Toxic Uncontaminated Work Areas (Hazard Category D).**

Within the CAMDS Site there are many areas inhabited by workers where non-toxic work operations are taking place. These areas are not under "engineering control" and have little or no potential for vapor contamination. These areas are not normally monitored for agent, however DAAMS only stations will be used to monitor areas adjacent to dilute agent storage areas, the SAF laboratory lunch room and agent contaminated hazardous waste Permitted storage areas (when waste is present) at the 12-hour WPL level

#### **4.2.4 Laboratory Work Areas**

Laboratory Work Areas shall be monitored with NRT and DAAMS stations in accordance with the Appendix A of Permit Attachment 16. Written approval is required from the Executive Secretary to reduce monitoring in any Laboratory Work area.

#### **4.2.5 Positive Pressure Areas**

The Control Module Operations area, Medical Module and Personnel Support Complex are the only areas which have the capability to switch the ventilation system to positive pressure when an agent alarm occurs in an area outside of engineering control. This allows personnel in these areas to perform work, without the need of a mask or protective clothing, if an upset condition exists.

Agent operations do not occur in these areas and with positive air pressure and there is no potential for vapor contamination.

#### **4.2.6 Filter Monitoring**

Multiple Bank Carbon Filter units (Appendix 18) provide negative pressure ventilation for potentially contaminated areas throughout the plant.

##### ***Six-Bank HVAC Filters***

***Filter mid-beds*** -The filter mid-bed locations (after banks one, two, and four) shall be monitored continuously with DAAMS for Mustard, GB, and VX at the WPL (12 hour) and for any other agents with which the carbon has been potentially contaminated.

***Filter Stack*** - NRT monitors shall be used to monitor for each agent being processed in the plant at the STEL (action level 0.5). DAAMS stations continuously monitor the HVAC Filter Stack for GB, VX, and Mustard at the WPL (12 hour).

##### ***CAMDS Laboratory HVAC Filters***

DAAMS shall monitor for GB, VX, GA, Mustard, and Lewisite between carbon banks 1 and 2, and at the Filter Stacks at the WPL (12 hour). The Filter exhaust Stacks shall be monitored with NRT monitors for GB and VX at the STEL (action level 0.5).

#### **4.2.7 MPF Stack**

The MPF Stack is continuously monitored, by both NRT monitors and DAAMS, at the SEL level for all agents being processed in the facility. The SEL provides an early indication of upset conditions, and must be accurately measurable in a timely manner. Staggered NRT monitors are required on the MPF stack for each agent. The two staggered NRT monitors shall have differing columns with two identical NRT monitors for backup. DAAMS are used as confirmation for any NRT alarms above the action level. A Waste Feed Cutoff for the MPF is initiated when an alarm at or above the 0.2 SEL action level is exceeded at the MPF stack.

##### ***MPF Stack NRT Monitoring Cessation***

Cessation of NRT monitoring on the MPF stack, while the furnace is running, can occur only under the following conditions:

1. All MPF operations have been completed (all burn baskets have been completely processed), and
2. The MPF stack has been continuously monitored with NRT monitors for a full 24 hours after completion of the campaign, without an alarm above the action level of 0.2 SEL.
3. Continuous monitoring with DAAMS tubes (4 hour SEL) is required for all agents processed in the completed campaign. DAAMS monitoring shall continue while the furnace is in operation or until the commencement of a new agent campaign and reconfiguration of the monitoring stations are required.

#### **4.2.8 Life Support System (LSS) Air Connects**

Life Support System (LSS) air is monitored with DAAMS at the WPL (2 Hours) level daily.

#### 4.2.9 Continuous Emission Monitoring System

The CAMDS Site Continuous Emissions Monitoring System (CEM), non-chemical agent stack monitoring, will be IAW Attachment 17 of this Permit.

#### 4.3 Perimeter Monitoring

The DCD perimeter requires historical monitoring with confirmation monitoring at the GPL level for all chemical agents stored in the storage yard. The CAMDS Site provides monitoring and analytical support for the perimeter monitoring. Perimeter Monitoring for a specific agent may only be discontinued if all of the recoverable neat agent, for that specific agent, has been demilitarized.

##### 4.3.1 Perimeter Stations

There are ten Perimeter Monitoring Stations running DAAMS tubes for), Mustard (12 hour). Each station has a primary, secondary, and tertiary DAAMS tubes for the Mustard agent monitored and are analyzed for agent at the GPL. Stations are numbered 901-908 and 910-911. Logbooks documenting all maintenance, inspections and monitoring activities shall be kept current at each station. The two igloos in Area 10 containing GA and Lewisite shall have filter systems placed on their air exhaust ducts. Headwall monitoring at the STEL is required at a minimum of twice a week for the agent contained in each igloo.

#### 4.4 AREA 10 Monitoring

The DCD Operations Support Directorate provides analytical support for Area 10. Area 10 personnel providing support for the CAMDS Site shall be in compliance with all analytical and training requirements of this Permit. Locations in Area 10 s supported by the DCD Operations Support are listed in Table 4.5.

Table 4-5: AREA 10 Areas Supported by the DCD Operations Support Directorate.				
Location Description	Station Number	Monitor Type	Agent Monitored	AEL Level
Lunchroom East BLDG 1810	1810LRE	DAAMS	GB, VX, and Mustard	WPL (12 hr.)
Lunchroom West BLDG 1810	1810LRW	DAAMS	GB, VX, and Mustard	WPL (12 hr.)
Equipment Clothing Aeration Room BLDG 1810	1810ECAR	DAAMS	GB, VX, and Mustard	WPL (12 hr.)
Smoke Shack BLDG 1810	1810SS	DAAMS	GB, VX, and Mustard	WPL (12 hr.)
Airlock BLDG 1810	1810AIRLOCK	DAAMS	GB, VX, and Mustard	WPL (12 hr.)
Break Room BLDG 1850	1850BR	DAAMS	GB, VX, and Mustard	WPL (12 hr.)
RDTE Solution Room BLDG 1850	1850RDTE	DAAMS	GB, VX, and Mustard	WPL (12 hr.)
Monitoring Room BLDG 1810	1810MR	DAAMS	GB, VX, and Mustard	WPL (12 hr.)

#### 4.5 Discontinuation of Monitoring in A, B and C areas

##### 4.5.1 A & B Areas

Monitoring in Category A and B areas, including LSS Air Monitoring, for past agent contamination may be discontinued when the airborne agent contamination for that area is less than 1.0 STEL over a 24-hour period, at a minimum temperature of 70° F, with the ventilation system operating at the approved flow rates. A confirmed agent reading at or above 1.0 STEL requires that the area must undergo additional decontamination. LSS hose monitoring for past agent may be discontinued if the hoses are less than 1.0 WPL.

#### **4.5.2 C Areas**

Monitoring in Category C areas for past agent campaign contamination may be discontinued when the Category C area has been less than 0.5 WPL<sub>(12-hour)</sub> for each agent being discontinued over a 24-hour period, with the adjacent A& B area ventilation systems operating at the approved flow rates, at a minimum temperature of 70°F. Monitoring data must be submitted to the Executive Secretary for approval prior to discontinuation of any agent monitor in a C area, with the exception that approval of the submitted monitoring data is not required before shutting off monitors in C areas with approved closure plans.

### **5.0 AGENT AIRBORNE EXPOSURE LIMITS (AEL) AND CATEGORY AREAS**

Airborne Exposure Limits (AEL), for monitored agents at the CAMDS Site, are compiled in Table 5-1. AELs are standards that are set by the CDC for chemical agent exposures. The AEL Federal Register references are as follows 1) 68 FR 58348 (October 9, 2003) for GA, GB and VX, 2) 69 FR 24164 for Mustard, and 3) 53 FR 8504-85077 (March 15, 1988) for Lewisite.

#### **5.1 AEL Definitions**

##### **5.1.1 Immediately Dangerous to Life and Health (IDLH)**

IDLH is the maximum concentration, in the event of respirator failure, where a person could escape, within 30 minutes, without a respirator and without experiencing any escape impairing (e.g. severe eye irritation) or irreversible health effects. IDLH levels are 0.1 mg/m<sup>3</sup> for GB/GA, 0.003 mg/m<sup>3</sup> for VX, and 0.7 mg/m<sup>3</sup> for Mustard. The Mustard IDLH is only based on non-carcinogenic effects.

##### **5.1.2 Short Term Exposure Limit (STEL)**

STEL is the maximum concentration to which unprotected chemical agent workers may be exposed to for up to 15 minutes, continuously. The STEL for GB/GA is  $1 \times 10^{-4}$  mg/m<sup>3</sup> with a limit of four daily exposures. The STEL limit for VX is  $1 \times 10^{-5}$  and the limit for Mustard is  $3 \times 10^{-3}$ . Only one exposure daily is allowed at the STEL for VX and for Mustard.

##### **5.1.3 Worker Population Limit (WPL)**

WPL is the average allowable concentration that an unmasked worker could be exposed to for an 8 hour workday, 40 hours per week for 30 years without adverse effects. The WPL for the CAMDS Site has been adjusted to reflect a 12-hour work shift. The 12-hour WPL for GB/GA is  $2 \times 10^{-5}$ , VX is  $6 \times 10^{-7}$ , and HD is  $2.7 \times 10^{-4}$ .

##### **5.1.4 General Population Limit (GPL)**

The GPL is an allowable 72-hour time-weighted average concentration for the general population. The limit applies to the entire population, including all ages and medical conditions. For GB, the GPL is  $1 \times 10^{-6}$  mg/m<sup>3</sup> for 24 hours, VX, the GPL is  $6 \times 10^{-7}$  mg/m<sup>3</sup> for 24 hours and for HD, the GPL is  $2 \times 10^{-5}$  mg/m<sup>3</sup> for 12 hours.

## 5.2 Other Non-health Based Emission Limits

### 5.2.1 Source Emission Limit (SEL)

Source Emission Limit was previously known as the Allowable Stack Concentration (ASC). SEL is a ceiling value that serves as a source emission limit, and not as a health standard. The SEL is used for monitoring the MPF stack. The SEL provides an early indication of upset conditions, and must be accurately measurable in a timely manner.

It should be noted when monitoring at the common stack, that because of the high temperature and moisture content of stack emissions, dilution control devices are used in conjunction with the NRT monitors and DAAMS. The SEL value for GB and VX is 0.0003 mg/m<sup>3</sup> and Mustard is 0.03 mg/m<sup>3</sup>.

<b>Table 5.1: Agent AEL levels.</b>						
<b>AEL Levels (mg/m<sup>3</sup>)</b>						
<b>Agent</b>	<b>GPL</b>	<b>WPL</b>		<b>STEL</b>	<b>IDLH</b>	<b>SEL</b>
<b>GA, GB</b>	1 x 10 <sup>-6</sup> (24 hours)	2 x 10 <sup>-5</sup> (12 hours)	6 x 10 <sup>-5</sup> (2 hours)	1 x 10 <sup>-4</sup> 15 minutes	1 x 10 <sup>-1</sup> ≤30 minutes	3 x 10 <sup>-4</sup>
<b>VX</b>	6 x 10 <sup>-7</sup> (24 hours)	6 x 10 <sup>-7</sup> (12 hours)	4 x 10 <sup>-6</sup> (2 hours)	1 x 10 <sup>-5</sup> 15 minutes	3 x 10 <sup>-3</sup> ≤30 minutes	3 x 10 <sup>-4</sup>
<b>H agents</b>	2 x 10 <sup>-5</sup> (12 hours)	2.7 x 10 <sup>-4</sup> (12 hours)	1.6 x 10 <sup>-3</sup> (2 hours)	3 x 10 <sup>-3</sup> 15 minutes	7 x 10 <sup>-1</sup> ≤30 minutes	3 x 10 <sup>-2</sup>
<b>Lewisite</b>	3 x 10 <sup>-3</sup> (12 hours)	3 x 10 <sup>-3</sup> (12 hours)	3 x 10 <sup>-3</sup> (2 hours)	3 x 10 <sup>-3</sup> 15 minutes	Not Established	3 x 10 <sup>-2</sup>

## 5.3 Hazard Category Areas

Table 5-2 defines the Hazard Category Areas at the CAMDS Site.

<b>Table 5-2: Hazard Category Areas at the CAMDS Site.</b>	
<b>Hazard Category Area</b>	<b>Activities</b>
<b>A</b>	The toxic processing area supported by the cascade ventilation system designated for probable liquid and vapor agent contamination (for example, munitions processing bay, toxic cubicle).
<b>B</b>	The toxic processing area supported by the cascade ventilation system is designated for possible vapor agent contamination only.
<b>C</b>	The nontoxic work area adjacent to Category A or B areas that are supported by the cascade ventilation system designated for possible low-level vapor agent contamination.
<b>D</b>	The nontoxic work area designation for areas considered uncontaminated.
<b>E</b>	The area designated for a positive pressure, filtered air environment.

## **6.0 STANDARD PREPARATION AND SOLUTION VALIDATION**

The standard solutions used for method and monitor calibration, along with the Chemical Agent Standard Analytical Reference Materials (CASARM) from which they are made, play an extremely important role in the quality assurance (QA) process. This section describes the procedures for preparation, handling, storage, and evaluation of standard agent solutions that are used by the CAMDS Site for calibration and spiking of monitoring and analytical equipment.

### **6.1 Chemical Agent Standard Analytical Reference Materials (CASARM)**

Edgewood Chemical and Biological Center (ECBC) supplies the CAMDS Site with Chemical Agent Standard Analytical Reference Materials (CASARM). The CAMDS Site laboratory shall keep a CASARM logbook and all pertinent data related to CASARMS.

#### **6.1.1 Neat CASARM**

The CASARM lots are high purity, neat chemical agents. These CASARMS are characterized by several analytical techniques. Neat CASARM are shipped by ECBC, in approved packaging, according to procedures that minimize the likelihood of chemical deterioration (i.e., in sealed ampoules under a blanket of dry inert gas and in the absence of light).

CASARM Quality Assurance Team (CQAT) provides users with quality control assurance documentation that specifies the certified purity of the CASARM, which are maintained at the CAMDS Site laboratory. CASARM shipments will be received at the CAMDS Site Laboratory (LAB). Representatives from the CAMDS Site and a DCD (Quality Assurance Specialist Ammunition Surveillance) (QASAS) will inspect the ampoules for any damage and ensure that information on the shipping documents and ampoules are correct. Neat CASARM will be stored at the CAMDS Site in accordance with (IAW) this CLMQCP, Standing Operating Procedure (SOP) LAB 66-00-01-04, or in the CASARM refrigerator in Area 10, DCD SOP TT-0000-L-147.

#### **6.1.2 Neat CASARM Dilution Operations**

The LAB at the CAMDS Site dilutes neat CASARM to provide the Research Development Test and Evaluation (RDTE) solutions IAW this CLMQCP and applicable SOPs (Appendix B). Chemical agent solutions diluted from CASARM are prepared in pesticide grade organic solvent. Adequate amounts of solvent from the same lot must be available to complete the solution. The Analytical Division will be responsible for the preparation of all stock solutions from CASARM.

An Agent Standard Log Book shall be established and maintained. The following information, at a minimum, will be recorded: Date of preparation, chemical agent used and purity, CASARM lot number, preparer's name, all tare weights and final weights, CASARM vial serial number, concentration of solution, solvent used (including manufacturer) grade, and lot number, analytical calculations, beginning and ending time of preparation, serial number of balance, any temperature variances above of 4 °C. When a chemical agent vial is removed from cold storage for use, the vial will be allowed to equilibrate to room temperature before it is opened. Any excess chemical agent remaining after standards preparation will be destroyed. Prior to use, all glassware, volumetric flasks, pipettes and/or burettes will be cleaned IAW SOP LAB 66-00-02-04.

#### **6.1.3 Procedures for Preparing and Storing Standard solutions**

Standard solutions are prepared by serial dilution of stock standard solution. Chemical agents VX, H, GA, Lewisite, and GB will be diluted with the appropriate solvent. Decontamination of

glassware and expired or unusable chemical agent solutions will be performed IAW SOP LAB 66-00-02-04.

All dilute CASARM will be stored at or below 4 °C. RDTE solutions shall be allowed to equilibrate to room temperature prior to use as this will help minimize contamination from condensation of atmospheric moisture, which would hasten agent degradation. Each time a vial of neat agent is opened for use, its headspace will be purged with a dry inert gas prior to replacement into cold storage. The time of all operations with CASARM outside of cold storage will be kept to a minimum. If parameters are not met the Division Chief and Accountable Officer and CASARM Quality Assurance Team (CQAT) shall be notified and an investigation shall occur which documents the impact to the sample quality.

The chemical agent GB, GA, H, VX, and Lewisite, Stock A standards, must be prepared semiannually; Stock A standards are prepared from CASARM. The Stock A concentration range of these standards will be greater than or equal to 120 micrograms per milliliter (µg/mL).

<b>Table 6- 1.</b>		
<b>RDTE Dilute Solution Preparation / Verification Frequency*.</b>		
<b>Agent</b>	<b>Concentration Range (ug/mL)</b>	<b>Minimum Preparation / Verification Frequency</b>
GB/GA (Stock A)	≥260	Every Six Months
GB/GA	< 100	Monthly
HD (Stock A)	≥2320	Every Six Months
HD	< 5 5 to 2300	Monthly Every two months
VX (Stock A)	≥ 120	Every Six Months
VX	< 100	Monthly
L (Stock A)	≥ 2320	Every Six Months
L (in methanol)	<2320	Weekly

**Records** – Records will be maintained in the laboratory for preparation of all working solutions. These records will include the agent name, the solution concentration, the solvent name, solvent grade, solvent supplier, solvent lot, the date prepared, the expiration date, the preparer's name, and (if applicable) the identity of the solution from which the serial dilutions were made. Standard vials will be labeled with the agent name, solution concentration, solvent name used to prepare the standards, and expiration date.

**Standard Q/C Crosscheck** - Chemical agent standards shall be crosschecked upon preparation, and whenever an operator suspects the validity of the standard concentration. Unsatisfactory concentrations will be turned in to the Laboratory Analytical Division and replacements obtained, when necessary. A Crosscheck Log shall be maintained in the laboratory.

**Standard Storage** - All standards will be stored and obtained IAW with this document and applicable SOP LAB 66-00-01-06. The CAMDS Site personnel store and obtain their solutions in Building 7085 during non-duty hours. Area 10 personnel store and obtain their solutions in Building 1850 during non-duty hours. Backup storage for standards will be located at the CAMDS Site laboratory.

#### **6.1.4 Dilute CASARMS**

The Stock A dilute solutions concentration ranges are listed in Table 6-1 for each agent. Actual concentrations of solutions may vary by no more than  $\pm 25\%$ . The Edgewood Chemical Biological Center's (ECBC) Chemical Quality Assurance Team (CQAT) shall provide documentation of the purity of the Stock A solutions and the shelf life of all standards. RDTE dilute Solutions shall be prepared/verified at the frequencies listed in Table 6-1.

## 6.2 Procedure of Validation of Standards

The LAB prepares dilute agent standards for the Monitoring Division to use for challenging and calibration of NRT monitors. Stock standards derived from neat CASARM will be verified by use of an internal standard for quality analysis. A separate verification standard procedure prior to use will verify working standards. New verification solutions will be prepared whenever new working solutions are prepared.

New stock standard concentrations will be verified, after preparation and monthly, by preparing a solution of stock standard with an internal standard. The Stock A calibration and the verification quality control standards will be analyzed using an internal standard procedure (SOP LAB 66-00-01-06) to determine if any degradation has occurred. This process will be performed at the time new standards are prepared and every month thereafter for the lifetime of the standards. The following are stock standard verification requirements:

1. Concentration results for new stock standards and monthly concentration verifications are acceptable if the Relative Response Factor (RRF) is within  $\pm 5\%$  of previous stocks initial RRFs and the Standard Deviation of the replicate injections (minimum 5) are less than 10%.
2. The chemical agent VX, GB, Mustard and GA standards will be prepared and validated in accordance with Table 6.1.
3. Stock A solution shall be compared against the new QC verification calibration standards using SOP LAB 66-00-01-06.
4. If the difference between the new calibration standard and the new QC verification standard is within 10% of each other the solutions will be validated. The RRF shall be determined by comparing the new working standard against the new verification standard. The following formula will be used to calculate the RRF:

$$\text{RRF} = (\text{Area}_{\text{working}})(\text{Conc}_{\text{verification}})/(\text{Area}_{\text{verification}})(\text{Conc}_{\text{working}})$$

**RRF = relative response factor**

**Area = instrument determined peak area**

**Conc = concentration**

5. If the difference between the compared results exceeds 10%, new standards will be prepared. The newly prepared standards will be subjected to the same treatment as shown above. If the second preparation of standards is not within  $\pm 10\%$ , the LAB manager will implement appropriate required corrective actions. All corrective actions will be documented.

## 6.3 Monitoring and Crosschecks

The Laboratory prepares dilute agent standards for the Monitoring Division to use for challenging and calibration of NRT monitors. The Monitoring Division will analyze the standards with the NRT crosscheck method below. If the NRT method shows a difference exceeding  $\pm 25\%$  between a new standard and the value determined a new NRT standard should be prepared.

Crosschecks may be performed as deemed necessary by monitoring personnel to verify solutions. The LAB provides chemical agent standards. The standards are provided in concentrations appropriate to the alarm level of the detector and the type of chemical agent monitored. Calibration and QC standards are prepared from the same working standards but placed in separate vials and labeled appropriately (two vial concept). Fresh vials of chemical agent standards must be obtained monthly. The person performing crosschecking procedures shall check standards each time new stock is prepared. Unsatisfactory concentrations will be turned in to the Laboratory Analytical Division and replacements obtained, when necessary. Chemical agent standards may be crosschecked whenever a monitoring operator suspects the concentration of the standard. A Crosscheck Log will be maintained to evaluate long-term trends in chemical agent concentration.

### **6.3.1 Agent Cross Check-Procedure**

The first consideration in performing a successful Agent Cross-Check is to ensure that the sampling equipment, normally an NRT unit, is in top working order. If the equipment is not running properly then the results obtained shall be considered inconsistent and the values shall be deemed unreliable. The crosscheck results must be repeatable.

*Perform the cross check for the CAMDS Site standards as follows:*

1. Inspect the NRT Pre-Concentrator Tube (PCT). The PCT must be clean and free of significant degradation. If the PCT is bad or in poor condition, replace it and fully condition (burn off) the new PCT before continuing.
2. Check the flow rates to insure there are no restrictions that could interfere with the NRT.
3. Begin the crosscheck procedure using an NRT configured to the agent of interest.
4. Re-challenge the NRT with the same agent standard to verify the 1<sup>st</sup> injection. Do not store or enter result.

Inject the NRT with the newest crosscheck standard using the same quantity as used in the previous standards. Compare results. The results must be within  $\pm 10\%$  of the previous standards.

### **6.4 Use of Commercially Available Standards**

Certified commercial chemical standards shall be maintained and stored in accordance with vendor-provided recommendations. If a certificate of analysis accompanies commercially available standards, further evaluation of the standard is not required; however, the standard must remain traceable to the certificate of analysis. If a certificate of analysis is not provided, the standard will be certified in accordance with vendor recommendations.

## **7.0 CERTIFICATION OF METHODS, INSTRUMENTS AND PERSONNEL**

The CAMDS Site Operation Support Directorate performs and documents certification and validation processes for operators, instruments and methods to confirm the analytical processes are acceptable for use. Acceptance testing of new analytical and monitoring equipment will be performed prior to field implementation and method certification for air monitoring methods will require completion of a successful P&A study and initial baseline study. Waste screening methods will require spike and recovery determinations (i.e., method detection limit (MDL) studies). Method certification will be required before the method can be used in field support operations. Method validation will be demonstrated through the continuous baseline study.

Assignable cause data attributed to instrument error not operator error may be removed from P&A and baseline studies.

## 7.1 Method Description

A method is a process that begins with the collection of a sample and is followed by analysis of the sample by an appropriate analytical technique. Method requirements specify the type of sampling media, airflow rates, collection time, the details of sample preparation, and the types and set points of instrumentation that will be used to analyze samples. Methods shall be placed under configuration control and critical parameters shall have identified tolerances that, when exceeded, will result in a “new” method.

Each monitoring and analytical method for sampling chemical agent material will be defined and documented. The following documentation shall be included with the method P&A Study.

**Application- Matrix** - Monitoring level and Target analyte(s).

**Sample collection** - Sampling device(s), flow rate, aspiration time, collection media (absorbent type, mesh size, and bed depth).

**Sample Preparation**- Sample analysis, instrument type, detector type and optical filter, configured instrument operational parameters (timing, temperatures, gas types, flow rates, pressures, peak parameters, error limits), column(s) (type, phase, phase thickness, length, and diameter), and carrier gas or mobile phase type.

## 7.2 P&A Method Certification

All methods shall successfully satisfy P&A study method certification requirements and/or waste method certification as required in Tables 7-1 and 7-2 before the method is allowed to support operations. All data from P&A studies, including method description as defined in paragraph section 7.2, shall be submitted to the CMA-Monitoring Office for review and concurrence before the method can support operations.

<b>Table 7-1: Type of P&amp;A and MDL Method Certification.</b>	
<b>Application</b>	<b>Type of Certification Required</b>
NRT Chemical Agent Methods	Class I
Historical Methods	Class I
Historical Perimeter Methods	Class I <sup>c</sup>
Confirmation Methods	Class I <sup>a c</sup>
Agent Waste Screening Methods	MDL Study
Waste Characterization Methods	MDL Study <sup>b</sup>
<b>Notes:</b> <sup>a</sup> A GC/MS connected to GC may have a Class II P&A certification for confirmation purposes only <sup>b</sup> Method Detection Limit (MDL) Study, as defined in 40 CFR Part 136, Appendix B <sup>c</sup> If class 1 criteria cannot be met, then other criteria maybe implement by approval from the Executive Secretary.	

**Table 7-2:  
P&A Method Certification Requirements.**

Type of Certification	Number of Operators	Number of Instruments	Number of Days <sup>a</sup>	Target Concentrations <sup>b</sup>	Total Number of Points	Criteria
<b>Class I</b>	2 or more	2 or more	4	1. Each operator shall challenge the instrument at the target concentration of 0.0, 0.5, 0.75, 1.0, 1.5, and 2.0 times the monitoring level (Z)  2. MPF stations require an additional target concentration of 0.2 Z.	48 MPF (56)	1. Target action level (TAL) is greater than the statistical calculated limit of quantitation (LOQ).  2. Uncertainty in found mass (UIFM) is less than or equal to $\pm 25$ percent.  3. Recovery at the monitoring level is within 75 to 125 percent.  4. For MSD methods, UIFM is less than or equal to $\pm 40$ percent.
<b>Modified Class 1 (Operators or Instruments has to equal one)</b>	1	1	4			
<b>Class II</b>	1	1	2	4 at 1.25Z, 8 each at 1.0 and 0.5Z (0.2Z) <sup>d</sup>	20	<ul style="list-style-type: none"> <li>All samples at 1.25, 1.0Z and 0.5 Z or (0.2Z) yield a positive response, as defined during method development.</li> </ul>
<b>Agent Waste Screening Methods Waste Characterization Methods</b>	1	1	1	Minimum of 7 replicate samples at the PQL	7	Recovery and precision defined by method development.
Notes: <sup>a</sup> Preferably consecutive days at a minimum within an eight day window. <sup>b</sup> The laboratory must request written approval from the CMA-Monitoring Office and the Executive Secretary to use different challenge levels. <sup>c</sup> Statistically determined outliers, not to exceed the square root of the total number of data points, may be excluded from the set. Assignable cause data shall be repeated with documentation as to why the data point(s) was repeated. <sup>d</sup> MPF stack confirmation DAAMS tubes would require a 0.2 Z challenge.						

When methods are identical, to include spiked mass, with the exception of the aspiration time and WPL monitoring level, only the longest sampling aspiration period shall require a P&A study. For example, a GB 4-hour WPL method with no respiratory protection and the GB 8-hour WPL method with no respiratory protection, assuming the methods are otherwise the same and the 1.0Z mass spikes are equivalent between the methods, only requires P&A certification of the GB 8-hour application.

When performing a P&A study, all sampling and analysis operations shall be performed exactly as set forth in the applicable analytical procedures. All P&A challenges will be in the form of quality plant (QP) samples. Method certification shall be performed with a representative sample matrix. Once a P&A study starts, all challenges will be part of the P&A study except for those during documented maintenance activities. All personnel performing method certification must be trained in the operation of the analytical equipment. For the purposes of this document, all fully quantitative analytical methods and monitors will be referred to as "Class 1" methods and monitors, and all qualitative methods and monitors will be termed "Class 2" methods.

### 7.2.1 Air Sampling Methods

For air sampling methods, the P&A challenges can be injected directly into the NRT instrument, distal end of the sample line, or sample collection media. If the P&A challenge is injected directly into the NRT instrument, this shall be performed at the beginning of the sample cycle and the sample line immediately reconnected (except for process areas where chemical material is present). For historical air methods, the P&A challenge shall be spiked onto the sampling media prior to air sample collection. For NRT confirmation methods, the P&A challenge shall be spiked onto the sampling media after (post-spike) air sample collection for maximum matrix effects.

Operators performing the study shall not be given access to the target concentration (TC) values until after the found concentration (FC) values have been reported. For quantitative methods, the concentration range must provide quantitative accuracy over the finite range of agent concentrations that the CMA laboratory is required to report. More stringent challenge levels may be specified by permit requirements, analytical method capabilities, and/or the alarm level set point.

### 7.2.2 CLASS I P&A Study

Class 1 methods are quantitative over a finite range of analyte concentrations. The Class I P&A study shall consist of analyzing ten agent challenges and two blank challenges on four (preferably consecutive) days by at least two operators and two instruments as specified in Table 8.3. The P&A will be performed on location in an environment consistent with the normal operation of the instrument/monitor.

**NRT Monitors** - For NRT monitor methods, the instruments shall be spiked in duplicate at 0.0 Z, 0.5 Z, 0.75 Z, 1.0 Z, 1.5 Z, and 2.0 Z. The Z value for the NRT methods will be the STEL, SEL, and IDLH values. MPF stack stations require an additional target concentration of 0.2 Z. Operators performing the study shall not be given access to the target concentration (TC) values until after the found concentration (FC) values have been reported.

**DAAMS** - For DAAMS methods, two samples each will be spiked at 0.0 Z, 0.5 Z, 0.75 Z, 1.0 Z, 1.5 Z, and 2.0 Z and aspirated IAW the proposed method. The Z value for the DAAMS methods will be the value for the airborne exposure limits for the WPL, GPL, and SEL monitoring levels. Samples used for confirmation P&A studies will be spiked after aspiration. All other DAAMS

P&A samples will be spiked prior to aspiration. MPF stations require an additional target concentration of 0.2 Z.

### **7.2.3 Multi-Agent Methods**

The definitions below shall define what is considered a “Multi-agent cocktail” for calibrating or challenging sampling instrumentation.

1. A single injection of a combined/mixed solution of VX and GB shall be considered a Multi-agent cocktail.
2. Also, the injection of two single standard solutions, one for GB and one for VX, simultaneously (within one NRT cycle), shall be considered a Multi-agent cocktail.

Multi-agent cocktails shall demonstrate analytical accuracy within  $\pm 25$  percent with 95 percent confidence at the monitoring/action level.

Multi-agent methods (Monitoring or Laboratory) (more than one agent monitored by the same instrument) shall undergo a P&A study using all analytes, with randomized analyte concentrations.

Multi-agent cocktails may be used to calibrate and challenge monitoring and analytical instrumentation for VX and GB.

Multi-agent methods, for GB and VX, are only permitted for use if GB CASARM Lot# GB-U-6184-CTF-N is used for making all GB standards (including multi-agent standards). Approval is required from the Executive Secretary before any other GB CASARM lot is used for making multi-agent cocktails.

### **7.2.4 Modified P&A Study**

In some cases, a Class I P&A study may not be feasible, due to lack of instrumentation, lack of trained analysts, or sensitivity issues associated with the chemical material. Written approval must be received from the CMA-Monitoring Office to perform a modified Class I P&A study. A Class I P&A study takes into account the variability between instruments and operators, thus demonstrating that the method can be performed by any similarly configured instrument and by any qualified and trained operator. A modified Class I study does not take into account the variability between instruments and/or operators. CMA discourages the use of modified Class I studies but will approve its use under special situations and with the following restrictions:

1. If a modified Class I P&A study is performed with only one instrument, then that instrument will be the only one approved to run the specified method.
2. If a modified Class I P&A study is performed with only one operator, then that operator will be the only one approved to run the specified method.

### **7.2.5 Class II P&A**

For Class II P&A study, a positive response shall be defined during method development and can be based on minimal signal height, minimal area counts, signal-to-noise ratios, or minimal percent recovery. Sample analyses for routine operations shall use the same definition of a positive response as used in the certification process.

The performance of Class 2 methods, which are intended to provide an indication of the presence of agent above a certain concentration level (as required to confirm or report values).

The P&A will consist of analyzing 12 aspirated samples (2-12 hours) consisting of 6 agent challenges at the Z level and 6 blank challenges. The Z level is determined by the alarm level of the NRT monitor. The challenges will be spiked after aspiration of the sample. The Z value represents the target confirmation level or the reportable level.

Class 2 methods will be certified for use if all of the samples spiked at the Z level have a minimum recovery of 75% and none of the 6 blanks have a positive response. A positive response is defined as a peak within the retention time window with a response of greater than 5 to 1 signal to noise ratio.

### **7.3 Performance of a Minimum Detection Limit (MDL) Study**

Following USAEPA 846 guidelines for performing MDL studies may also certify methods. MDL studies consist of analyzing 7 replicate spikes at the Practical Quantification Limit (PQL). The accuracy and precision is determined by performing the analyses of the seven replicate spikes. An acceptable MDL study is the calculated MDL value and shall be at least 3 times lower than the PQL.

### **7.4 Significant Changes in Methods or Monitoring Task**

The items listed below are significant changes in the method/monitoring tasks and require the performance of a new P&A study as described in Section 7.0.

1. A change in the volume of the air sample collected only if volume collected theoretically contains an amount of agent that is less than the lowest standard or greater than the highest standard used during the original P&A study based on the mass of agent equal to the Hazard Level (HL).
2. A change in the type of media used to collect the sample.
3. A change in the type of stationary phase in a GC column.

Method certification data are quality records and will be readily accessible. Organizations having performed their P&A and certification testing will maintain copies of the relevant P&A and certification data and results.

#### **7.4.1 CMA Statistical Program**

Data generated from a Class I P&A study shall be entered into the CMA mandated statistical program. For multi-agent methods, the program will be used to determine results for each analyte separately. Pooling all 4 days of generated data, into a single group, and performing a linear regression analysis of the TC versus the FC for the data population shall evaluate the results. The program shall calculate limit of quantification, uncertainty of found mass, and percent recovery. Statistically determined outliers, not to exceed the square root of the total number of data points, may be excluded from the set. Assignable cause data shall be repeated with documentation as to why the data point(s) was repeated.

### **7.5 Certification of Additional Instruments and Operators**

Additional operators and/or instruments will require an additional P&A study to become certified. If more than one operator and /or instrument is used for the modified P&A study the component

containing the variable (e.g., two operators, one instrument) certification of additional operators would be performed by calibrating and analyzing three blind QC solutions but certification of additional instruments would require a new P&A study.

## **8.0 PERFORMANCE OF THE INITIAL BASELINE STUDY**

Initial 28 day baseline study demonstrates the readiness of the monitoring system to support operations. No method shall be allowed to support operations until it can successfully satisfy the initial baseline study method certification requirements (Table 8-1). During this period (or sooner), all sampling lines shall demonstrate a recovery of  $\pm 40$  percent of the TC (at the applicable monitoring level) when challenged at the distal end (defined as the end of the sample line where the sample enters the line).

If an initial baseline study is failing, the study can be extended, as improvements are made and corrective actions take effect, shifting the 28-day window accordingly. A minimum of 20 days of data in a 28-day period is required for each initial baseline study. Excluding days from the study is only allowed if construction/systemization activities or other operations prevented the daily challenge or if a challenge is excluded due to documented assignable cause (for example, use of the wrong standard).

**Table 8-1: Initial and Continuing Baseline Performance Criteria.**

<b>NRT</b>	<b>Challenges/ QP Sample</b>	<b>Performance Standard for Each Analyte</b>
NRT Monitor	Daily, each station <sup>(a)</sup>	A statistical response rate at the alarm level $\geq 95\%$ for each station and a first challenge pass rate $\geq 75\%$ .
NRT Monitor – MPF Stack	Six times daily, each station <sup>(a)</sup>	Statistical response rate at alarm level $\geq 95\%$ for each station and first challenge pass rate $\geq 75\%$ for each station.
NRT Monitor - Process Areas	Daily, each station <sup>(a)</sup>	Either challenge pass rate $\geq 75\%$ for each station.
NRT Monitor - Industrial Chemicals NRT Monitor - Mobile Station <sup>(c)</sup>	Daily Each Station <sup>(a)</sup>	Either challenge pass rate $\geq 75\%$ for each station.
	1. Initial: One for each station 2. Continuing: stations every 4 to 5 hours and at the end of the workday or operation.	QP FC is $\pm 25\%$ to TC
<b>DAAMS Tubes</b>		
Historical and Other Class 1 Methods	Each method daily, rotating stations with each station challenged at least once every 28 days. At the MPF stack a historical QP sample and a corresponding “A” tube will be collected for analysis once every 4 hours for each agent being monitored.	Challenge/QP FC $\pm 40\%$ to TC at a Reporting Level/Action Level of 0.50 Z Statistical response rate at reportable limit $\geq 95$ percent for each method. Data for all stations shall be pooled together by method.
Historical – Perimeter	Each method daily, rotating stations	Challenge/QP FC $\pm 40\%$ to TC at a Reporting Level/Action Level of 0.50 Z Statistical response rate at reportable limit $\geq 95$ percent for each method. Data for all stations shall be pooled together by method.
DAAMS Confirmation – MPF Stack NRT monitors and Process Effluents	Daily each method and each station <sup>(a)</sup> At the MPF stack a historical QP sample and a corresponding “A” tube will be collected for analysis once every 4 hours for each agent being monitored.	Challenge/QP FC $\pm 40\%$ to TC at a Reporting Level/Action Level of 0.20 Z Statistical response rate at reportable limit $\geq 95$ percent for each method. Data for all stations shall be pooled together by method.
Confirmation of NRT Monitor – Agent	Each method daily, rotating stations with each station challenged at least once every 28 days	Challenge/QP FC $\pm 40\%$ to TC at a Reporting Level/Action Level of 0.20 Z Statistical response rate at reportable limit $\geq 95$ percent for each method. Data for all stations shall be pooled together by method.
Confirmation of NRT Monitor – Industrial Chemicals	Once per month	In Accordance with manufacturer specifications.
<b>Sample Lines</b>		
All monitoring sample lines	Once every sixty days $\pm 5$ days (See Section 16.0)	Initial acceptance testing before agent use shall show a transmission efficiency of $\geq 75\%$ Continuing Challenge/QP FC $\pm 40\%$ to TC at an action level of 0.50 Z
<b>Notes:</b> (a) Where multiple near real time (NRT) monitors are used to sample the same location, each instrument will be considered its own station. A minimum of 12 hours is required between daily challenges.		

Initial baseline studies shall demonstrate the readiness of the monitoring system to support operations. All data from the initial baseline studies shall be submitted to the CMA-Monitoring Office for review and concurrence. Method validation will be demonstrated by the continuing baseline study. A continuing baseline study (minimum of 4 consecutive weeks of data) shall be performed following the initial baseline in accordance with section 8.1. Table 8-1 summarizes the type of stations and challenges required for conducting initial and continuing baseline studies.

### **8.1 Continuing Baseline Study**

The continuing baseline study validates the long-term performance of the monitoring system and starts immediately after successful completion of the initial baseline.

Continuing baseline requirements:

1. Each NRT station is an independent baseline study. Passing or failing the continuing baseline will be on a per station basis and not on a global basis.
2. Each historical and confirmation method is an independent baseline study. Passing or failing the continuing baseline will be on a per method basis.
3. Failing to meet the performance standards described in Table 8-1 will require corrective action and additional QPs until the problem is resolved. The problem and corrective action shall be documented in a Corrective Action Report.
4. For historical or confirmation stations failing a QP challenge, corrective actions will be required and daily QP challenges will continue until the problem is resolved and a passing QP challenge is obtained for that station. These diagnostic QP challenges shall not be included in the baseline studies, but shall be reported as part of the Corrective Action Report.
5. All sampling lines, except those used to monitor toxic process areas, shall be tested at a minimum of every 2 months to demonstrate a recovery of  $\pm 40$  percent of the TC (at the applicable monitoring level) when challenged at the distal end (see Section 16.0).
6. Certified operators shall participate in the continuing baseline study.
7. All continuing baseline data shall be transmitted to the CMA mandated statistical program every 2 weeks.

### **8.2 Alternative Baseline Studies**

Table 8.2 listed when Alternate Baseline studies may be used and the initial and continuing monitoring requirements. Written approval shall be required from CMA before any Alternate Baselines are implemented at the CAMDS. All Alternate baseline and continuing baseline data shall be sent to CMA for review. Alternate baselines may not be implemented on the MPF Stack or HVAC monitoring systems. When performing the baseline studies all sampling and analysis operations shall be performed as set forth in the applicable analytical or monitoring procedures unless more restrictive procedures are required in Table-8-2. Where multiple near real time (NRT) monitors are used to sample the same location, each instrument will be considered its own station

**Table 8-2:  
Initial and Continuing Alternate Baselines: Performance Criteria.**

Type of Monitoring Operation	Initial Baseline /Challenges/ QP Sample	Performance Standard for Each Analyte
<p><b><u>Short-term Operations NRT and DAAMS Monitoring.</u></b> 1-3 day operations in areas of the plant not operating under an approved closure plan in which the monitoring systems are taken offline (calibrate, service or challenge mode) at the end of the workday. Monitors that are shutoff shall have to be calibrated and the line re-challenged when monitors are turned back on.</p>	<p><b><u>Initial:</u></b> 1. Calibrate NRT (required on initial start-up of NRT). 2. Line Challenge (<math>\pm 40\%</math>) NRT and Historical/Confirmation DAAMS Lines (required at start-up). <b><u>Continuing:</u></b> 1. Beginning of workday, challenge the NRT and continue to challenge NRT every 4 hours (<math>\pm 30</math> minutes) and at the end of each workday. DAAMS tubes will be pulled and analyzed very four hours and at the end of the work day. 2. If operations logistics require the project to exceed 3 days then operations will discontinue until a full 28 day baseline can be completed or an approval is granted by the Executive Secretary to complete the operation using a 3-day baseline criteria.</p>	<p><b><u>NRT</u></b> Statistical response rate at 0.5 Z alarm level <math>\geq 95\%</math> for each station and first challenge pass rate <math>\geq 80\%</math> for each station. <b><u>Historical and Confirmation DAAMS</u></b> <b><u>Historical:</u></b> Challenge/QP FC <math>\pm 40\%</math> to TC at a Reporting Level/Action Level of 0.50 Z. Statistical response rate at reportable limit <math>\geq 95</math> percent for each method.. <b><u>Confirmation:</u></b> Challenge/QP FC <math>\pm 40\%</math> to TC at a Reporting Level/Action Level of 0.50 Z. Statistical response rate at reportable limit <math>\geq 95</math> percent for each method.</p>
<p><b><u>NRT and DAAMS Closure Operations Monitoring.</u></b> 1. Monitoring systems are monitoring continuously and are not taken offline (calibrate, service or challenge mode) at the end of the workday.</p>	<p><b><u>Initial:</u></b> 1. Calibrate NRT (required at initial start-up of NRT). 2. Line Challenge (<math>\pm 40\%</math>) NRT and Historical/Confirmation DAAMS Lines (required at start-up). 3. Consecutive 3-day Baseline at each NRT station. The NRT 3-day baseline shall be comprised of three daily challenges for each station (at least 4 hours between challenges) per day. <b><u>Continuing:</u></b> Challenge once a calendar day. There shall be a minimum of 12 hours between daily challenges for each station.</p>	<p><b><u>NRT</u></b> Statistical response rate at 0.5 Z alarm level <math>\geq 95\%</math> for each station and first challenge pass rate <math>\geq 80\%</math> for each station. <b><u>Historical and Confirmation DAAMS</u></b> Each method daily, rotating stations. <b><u>Historical:</u></b> Challenge/QP FC <math>\pm 40\%</math> to TC at a Reporting Level/Action Level of 0.50 Z. Statistical response rate at reportable limit <math>\geq 95</math> percent for each method.. <b><u>Confirmation:</u></b> Challenge/QP FC <math>\pm 40\%</math> to TC at a Reporting Level/Action Level of 0.50 Z. Statistical response rate at reportable limit <math>\geq 95</math> percent for each method.</p>
<p><b><u>Short-Term Closure Operations Monitoring</u></b> 1. 1-3 day Closure Operations where the monitoring systems are taken offline (calibrate, service or challenge mode) at the end of the workday. Monitors that are shutoff shall have to be calibrated and the line re-challenged when monitors are turned back on.</p>	<p><b><u>Initial:</u></b> 1. Calibrate NRT (required at initial start-up of NRT). 2. Line Challenge (40%) NRT and Historical/Confirmation DAAMS Lines (required at start-up). <b><u>Continuing:</u></b> 1. Beginning of workday, challenge NRT and continue to challenge NRT every 4 hours (<math>\pm 30</math> minutes) and at the end of each workday. DAAMS tubes will be pulled and analyzed very four hours and at the end of each workday. 2. If operations logistics require the project to exceed 3 days then operations will cease until 3 days of consecutive data, which satisfy the requirements of a successful 3-day baseline, are completed. One daily challenge shall be done after completion of the 3-day baseline.</p>	<p><b><u>NRT</u></b> Statistical response rate at alarm level <math>\geq 95\%</math> (0.5 alarm level for each station and first challenge pass rate <math>\geq 80\%</math> for each station). <b><u>Historical and Confirmation DAAMS</u></b> <b><u>Historical:</u></b> Challenge/QP FC <math>\pm 40\%</math> to TC at a Reporting Level/Action Level of 0.50 Z. Statistical response rate at reportable limit <math>\geq 95</math> percent for each method.. <b><u>Confirmation:</u></b> Challenge/QP FC <math>\pm 40\%</math> to TC at a Reporting Level/Action Level of 0.50 Z. Statistical response rate at reportable limit <math>\geq 95</math> percent for each method.</p>

### 8.3 Baseline Recertification

Cessation of monitoring activities (taking equipment offline and/or stop doing QPs) greater than 60 days for any NRT station, historical method, or confirmation method shall require repeating the initial baseline study and re-establishment of the continuing baseline for that particular station and/or method. All the initial baseline requirements shall apply.

Cessation of NRT monitoring activities for less than or equal to 60 days will require recertification in accordance with Table 8-3. Cessation of historical and confirmation methods less than or equal to 60 days does not require recertification. MPF stack recertification data shall be submitted to the CMA mandated statistical program. Instruments or monitors, that fail to meet these requirements, will be tagged for repair or replacement. When instruments undergo maintenance (not programmed maintenance) or are in continuous use with another agent for six months recertification is not required. If calibration is not within tolerance, further maintenance will be performed and recertification is required.

<b>Table 8.3: Continuing Baseline Recertification Requirements.</b>			
<b>Application</b>	<b>Number of Days Suspended</b>	<b>Number of Challenges<sup>a</sup> Required</b>	<b>Performance Standard</b>
NRT	1 to 10	1	±25 percent of TC
NRT – MPF Stack	1 to 10	6 daily <sup>b</sup>	All challenges ±25 percent of TC
NRT	11 to 30	2 <sup>c</sup>	All challenges ±25 percent of TC
NRT – MPF Stack	11 to 30	6 daily for two days <sup>b</sup>	All challenges ±25 percent of TC
NRT	31 to 60	4 <sup>c</sup>	All challenges ±25 percent of TC
NRT – MPF Stack	31 to 60	6 daily for five days <sup>b</sup>	Statistical response rate ≥ 95 percent
Notes: <sup>a</sup> All challenges at 1.0Z of monitoring level. <sup>b</sup> Challenges shall be 4 hours apart. <sup>c</sup> Successive.			

## 9.0 PERSONNEL TRAINING AND CERTIFICATION

Laboratory and monitoring personnel shall be trained in the operation of the equipment and methods prior to conducting method certification. Upon successful certification of the method the operator is certified for the particular method.

## **9.1 CAMDS Laboratory Training**

The Laboratory Analytical Division will maintain description of duties for all LAB job positions and ensure that the individuals assigned to a job position have the requisite experience and training that are consistent with the descriptions of duties for the job position.

Job descriptions and the training plan for government employees will be maintained and filed in the Laboratory Analytical Division. A copy of the job descriptions and Certification Plans for the Laboratory Analytical Contractor personnel shall be maintained in the Laboratory Analytical Contractor Office. The Analyst Certification Plan is divided into two phases.

Operations Support Director and Laboratory Analytical Manager shall be responsible for certifying government and contractor personnel respectively. There are two phases to the Laboratory training plan.

### **9.1.1 Phase One**

Phase One includes RCRA Hazardous Waste training and Chemical Surety, which encompasses an initial course and an annual refresher course. This phase of the training process is required of all Laboratory Analytical Division personnel and consists of the following general orientation, toxic and hazardous waste training:

#### ***General Orientation Training***

1. Site description.
2. Chemical agents GB, VX, GA, HD, and L.
3. Process overview.
4. Site safety procedures.
5. Site security and surety procedures.
6. Emergency conditions, alarms, and procedures.

#### ***Toxic Area Training***

1. Toxic areas.
2. Protective clothing.
3. Entry and exit procedures.
4. Decontamination techniques.
5. Emergency medical treatment.

#### ***Hazardous Waste Training***

1. Hazardous Communications.
2. Hazardous Materials.
3. Toxic Chemical Safety.

Upon completing each topical area, personnel must take a test to demonstrate understanding of the subject matter presented. A topic that is failed may be repeated a second time. A second failure disqualifies a person from working in the Laboratory.

### **9.1.2 Phase Two**

Laboratory Academics and Practical Application Training comprises Phase Two. This training is a requirement for chemists and technicians working in the Laboratory. The Laboratory Analytical Manager may elect to exempt personnel from any training in this phase, based on documented course work in this area and/or demonstrated ability. Personnel training will be based on current and future analytical tasks and number of trained personnel available. Phase Two training includes the following:

1. Laboratory essentials.
2. Analytical fundamentals.
3. Agent Laboratory essentials.
4. Laboratory instrumentation.
5. Laboratory QC.
6. Sample preparation.

### **9.1.3 Written Exam**

The trainee will be shown proper analytical techniques using the procedure with which the trainee is qualified. After the trainee has become familiar with the analysis and relevant calculation, the trainee will qualify by passing a written examination based on the principles and techniques shown for each method in this phase. Additionally, the trainee will be required to pass the demonstration of method and techniques as outlined for each method in this section. If the trainee demonstrates an acceptable level of expertise, the qualifier will sign the trainee's qualification form. If the trainee fails, a retest may be given. If the trainee fails a second time, a second retest may not be given for 30 days.

### **9.1.4 Analyst Certification Records**

The LAB will maintain the analyst certification records. Files containing original documentation of analyst certification will be maintained for each employee. Analyst certification files will be made part of the 40-year record.

### **9.1.5 Analyst Proficiency Review**

The competency of analysts performing Class 1 methods will be evaluated semi-annually. In the course of a year, all certified operators using all the Class 1 methods for which the individual has been certified shall be required to analyze blind samples. An individual who fails to meet the certification criteria for a method(s) must pass a certification test for the method(s) as described in Section 9.1. Records of personnel certifications shall be maintained at the CAMDS Site.

An analyst who performs an evaluation test using any particular monitoring technique (e.g. NRT, DAAMS) for a given agent is not required to be evaluated using that technique at higher monitoring levels with the same agent.

### **9.1.6 Analyst Recertification**

An individual who has not used a Class 1 instrument for two calendar months will read the appropriate procedure or SOP, document that it has been read, and analyze duplicate samples of 0.2 Z, 1.0 Z, and 1.5 Z. The analyst is re-certified if all of the found concentrations are within  $\pm 15\%$  for QLs or  $\pm 25\%$  for NRT QPs and  $\pm 40\%$  for DAAMS QPs.

## **9.2 Training & Certification of Monitoring Operators**

The Monitoring Division shall have a written training/certification plan for employees, of the Monitoring Division, within 45 days of the acceptance of this Permit modification, for approval by the Executive Secretary. Upon completion of training and experience, copies of all certificates and information will be added to the employee's permanent files and kept on file with the training coordinator in the office of the Director of Resources. Initial training will consist of:

1. Chemical Surety
2. Hazardous Communication
3. Hazardous Waste
4. Hazardous Materials
5. Toxic Chemical Safety
6. Emergency Response to Hazardous Spills
7. Other job specific requirement, i.e., on-Site or off-Site NRT training.

Refresher training will also be taken yearly by the employee for each of the above subjects, or more often as required by applicable army regulations or this Permit.

The operator certification plan shall prescribe the policies, rationale for the certification, organizational responsibilities, and criteria to be applied to the certification of personnel in the monitoring support operations at the CAMDS Site. This plan is applicable to all government or contractor technicians and operators who support monitoring operations. This plan will provide a high level of confidence that an operator, certified IAW this plan, will generate analytical data of an accuracy and precision consistent with the limitation of the methodology.

Certification of operators, of chemical agent detectors, requires that operators read all pertinent monitoring related documentation, have on the job training as required in the plan, and demonstrate the capability to perform a P&A evaluation for the detector. Documentation must be maintained on file that includes the raw data used for the evaluation. Certification also requires that the operator demonstrate a level of knowledge commensurate with the particular task. Such knowledge is necessary in order to properly ensure the protection of the workers and the environment while also providing valid documentation on the containment of chemical agents to meet regulatory requirements. This section of this document sets forth the requirements necessary to administer this program. The, Monitoring Division Chief, shall review and sign the results of training and certification indicating successful completion of training.

Operators who leave the Monitoring Division shall recertify when rehired or upon returning to the Monitoring Division, using the same criteria as newly certified operators.

A two level certification process will be required for certification of monitoring operators depending on the job functions of the operators.

### **9.2.1 Level I Certification Process**

Level I certification requires training for operators in the operation and maintenance of Class I and Class II monitors. A certification assessment shall be required, to test the proficiency of each operator. Testing shall include proficiency in challenging sample lines with NRT monitors or DAAMS tubes. All training and certification assessments completed shall be documented and the records kept by the Monitoring Division.

Certification assessment, for Class I Monitors requires candidates to successfully complete all training requirements listed below for each method and certification testing. The Monitoring Division Chief or his designated representative shall verify successful completion of each task. The designated representative shall be a Class I Certified Operator with records on file documenting certification and training for the specified method.

#### ***NRT Operators***

In addition to RCRA and required Army training, there are two methods of training required before employee can be tested and certified as NRT Monitoring Operators. Training shall include the attendance of an applicable training course, and on the job instruction by a Certified Monitoring Operator (minimum of 30 days). Successful completion of a written test and a performance evaluation are mandatory. The applicable Division Office maintains training records. Training shall include applicable Standard Operating Procedures (SOP) and general training on NRTs, including and overview of systems components of the NRT modules, front panel module, GC module, card cage, power supply, transformer module, chassis module, cable module, gas specifications, sample pump, recorder module, accessories module, safety, quality control requirements, documentation of calibration, and QC Challenges are required.

#### ***DAAMS Operators***

DAAMS operator training shall require competency in the following procedures; 1) visual inspection of DAAMS, 2) labeling (Bar Code system if applicable), 3) sample line connection, 4) verification of flow checks, 5) VX conversation pad requirements, 6) dealing with broken DAAMS tubes, 7) proper documentation, 8) the setting of timers, 9) visual inspection of the equipment (i.e., pump, tubing, electrical cord) and replacement of NOX filters.

#### ***RTAP Operator***

RTAP (Vehicle Only) operator certification shall be required. Operators must have completed the CAMDS Site RTAP Monitoring Operator certification requirement. The, Monitoring Division Chief, shall review and sign the results of training and certification indicating successful completion of training.

### **9.2.2 Recertification of Operators**

#### ***Class 1 Methods/Monitoring Tasks***

Recertification of monitoring personnel is instrument based not method based. An individual who has not used a Class 1 instrument or monitor for six months will be trained and certified prior to using the instrument or monitor. The operator shall be required to review and document that they have read the appropriate sections of CLMQAP, the Monitoring Plan and monitoring SOP's. In addition, the monitoring operator shall inject, an NRT monitor, with QP challenges, at 0.5Z, 1.0Z and 1.5Z (an additional challenge at 0.2Z is required on the MPF stack NRT monitors), once per day for two consecutive days and perform one successful line challenge.

All injection challenges must be within  $\pm 25\%$  to successfully complete certification requirements. Operators performing the study shall not be given access to the target concentration (TC) values until after the found concentration (FC) values have been reported. If one of the six-sample challenges in the personnel certification is not within the acceptable limits, an additional three-sample-set challenge will be performed. These three-sample challenges will be within acceptable limits or the entire 6-sample recertification test must be repeated.

An individual who has not used a Class 1 monitor for two calendar months will read (documented) the appropriate procedure or SOPs.

Operators maintaining DAAMS stations shall have a period of on the job training and will complete an operational checklist prior to operation if they have not performed this task for more than 60 days (documented).

RTAP (Vehicle Only) operator recertification shall be required according to specifications in the Training Plan if they have not performed this task within 60 days.

### **9.2.3 Operator Proficiency Review**

The proficiency of the operator performing Class 1 methods shall be evaluated with documentation monthly and quarterly (at a minimum). The Quality Assurance Coordinator (QAC) will perform this evaluation based upon the results of the monthly performance charts provided to him by the Monitoring Division. The Monitoring Division will provide charts to the QAC no later than ten working days after the end of each month. After analyzing the results, the QAC will pass the results to the Monitoring Division Chief for review and corrective action if required.

## **10.0 CALIBRATION OF ANALYTICAL INSTRUMENTS AND MONITORS**

Calibration involves analytical instruments and monitors, for which the instrument response for unknown samples are compared to a calibration curve generated by a range of samples whose concentrations, are known. The primary purpose of this calibration is to establish a correlation between instrument response and analyte concentration in the medium to be analyzed. A secondary purpose of this calibration is to verify that the instrument is performing properly.

DCD has established a calibration coordinator for the installation. The calibration coordinator has unit coordinators assigned by each directorate to assist with the program. The Test Measurement and Diagnostic Equipment (TMDE) group, at Dugway Proving Grounds, conducts periodic calibration. DCD has written procedures in *Quality Plan #8, Control of Monitoring and Measurement Devices*. The coordinator will be told of any unusable equipment. If the equipment is or has been chemically contaminated, the item will be considered as hazardous waste and will be disposed of accordingly.

The CAMDS Site monitoring Unit Calibration coordinators will be responsible for the following:

1. Maintaining the location and status of TMDE of the organization.
2. Providing copies of the calibration recall lists to responsible personnel to inform them of TMDE calibration due dates.

3. Ensuring that each item of equipment is labeled, marked, or otherwise identified to indicate its calibration status.
4. Maintaining a list of equipment submitted to the TMDE.
5. Keeping supervisors informed of TMDE calibrations that are required.

#### **10.1 NRT Monitors, DAAMS, and Dilution Air Flow Controllers**

Mass Flow meters for NRT monitors, Dilution Air Flow Controllers and Mass Flow meters (portable) used to measure airflow on DAAMS systems shall be calibrated by actual volumetric flow at site-specific ambient conditions by trained CAMDS Site monitoring personnel (training and calibration documented).

#### **10.2 Frequency of Calibration: DAAMS Class 1 Laboratory Method**

Calibration of the DAAMS GC will consist of analysis of a minimum of three calibration standards covering the range of 0.2 to 1.5 (SEL, STEL), 0.5 to 2.0 (WPL, GPL, all others) times the High Alarm Level (HL) with at least one standard below and one standard above the HL. Calibration will be done at a minimum of every seven days.

To spike a sorbent tube, the required volume of a standard solution of the agent of interest is injected onto the sorbent bed and then ambient air is drawn through the tube for a brief period to remove solvent. After the sorbent tubes have been spiked, they are analyzed in the usual manner.

The peak responses and the retention times reported by the GC for the calibration samples shall be electronically recorded and stored in the 40-year record. The following criteria must be met for a successful calibration.

1. The regression parameters and the variability of the retention times will be compared to those obtained from previous recent calibrations to ensure that no sudden change in instrument response has occurred.
2. The absolute value of the correlation coefficient must be greater than 0.995.

#### **10.3 Laboratory System Acceptance Criteria**

System acceptance criteria shall meet the minimum response requirements before calibration may continue. Systems that fail to meet this criterion will undergo corrective action. Specific criteria can be found in SOP LAB 66-00-00-16, LAB 66-03-01-01, and Laboratory Instrument calibrations are performed when any of the following conditions occur:

1. Seven or more days have passed since the last calibration.
2. No FPD calibration exists.
3. Significant maintenance is performed on the GC-FPD.
4. When CCVs fail to meet the requirements found in section 14.10.

Agent standards used for instrument calibration will be from a different set than agent standards used for spiking QL or QP samples. Chemical agent calibration will cover the mass range of 0.2 to 1.5 (SEL, STEL), 0.5 to 2.0 (WPL, GPL, all others). The Z values are listed in Table 10-1. Calibration curves shall be generated, depending on the configuration of the GC according to Table 10-1. Each of these calibration curves will consist of at least five spiked samples. One spike will be analyzed for each calibration point, except the low spike point, which will be

analyzed in triplicate (three spiked samples at the same concentration). A fourth low datum point may be used. If four data points are used and the average recovery of all calibration points is not within  $\pm 15\%$  of the target, or if one of them is not within  $\pm 5\%$  of the retention time averages, then one of the four identical spikes will be rejected.

The solvent spike and the calibration spike will be directly injected into the DAAMS tube. The DAAMS samples will be analyzed using instruments that have been calibrated to cover the monitoring level range of 0.2 to 1.5 (SEL, STEL), 0.5 to 2.0 (WPL, GPL, all others).

Linear regression curves will be generated for chemical agents GB, VX, L, and GA. The DAAMS GC-FPD calibration curves for chemical agent H are nonlinear and require a different calibration fit. Calibration curve acceptance must result in a correlation coefficient ( $r$ ) for chemical agents GB, VX, and H of 0.995 and  $r$  for chemical agents GA and L of 0.990. The three low spike samples of each calibration range must have a relative standard deviation of  $< 25\%$ . The retention times of all data points must be within  $\pm 5\%$  of the average of all calibration retention times. The calibration points are described in Table 10-1.

**Table 10-1: GC-FPD/ Calibration Points.**

AGENT METHOD	HAZARD LEVEL (Z)	HAZARD LEVEL CONC. (mg/m <sup>3</sup> ) (Z)	SAMPLE TIME (min)	FLOW RATE (L/min) <sup>(2)</sup>	QPs/24 (HOURS)	1.0 Z QP SPIKE <sup>(7)</sup> (ng)	THREE POINTS <sup>(3,4)</sup> (ng)	ONE POINT <sup>(3)</sup> (ng)	ONE POINT (ng)
GB DAAMS	WPL	2.00E-05	720.00	0.50	2	7.2	3.6	7.2	14.4
GB DAAMS	WPL	1.00E-04	120.00	0.50	2	6.0	3.0	6	12
GB DAAMS	SEL	3.00E-04	240.00	0.50	4	6.000	0.220	1.100	1.6
GB DAAMS	GPL	1.00E-06	1440.00	0.50	2	0.72	0.36	1.44	1.6
GB NRT <sup>(5)</sup>	STEL	1.00E-04	1.83	1.00	2	1.100	0.036	0.180	0.270
VX DAAMS	WPL	6.00E-07	720.00	0.50	2	0.216	0.108	.216	0.432
VX DAAMS	WPL	4.00E-06	120.00	0.50	2	0.24	0.12	0.24	0.48
VX DAAMS	SEL	3.00E-04	240.00	0.50	4	3.6	0.720	3.600	5.400
VX DAAMS	GPL	6.00E-07	1440.00	0.50	2	0.432	0.216	.432	0.864
VX NRT <sup>(5)</sup>	STEL	1.00E-05	3.83	1.00	2	0.038 <sup>(8)</sup>	0.0076	0.038	0.057
H DAAMS	WPL	2.7E-04	720.00	0.5	2	97.2	48.6	97.2	194.4.
H DAAMS	WPL	1.6.00E-03	120.00	0.50	2	96	48	96	192
H DAAMS	SEL	3.00E-02	240.00	0.50	4	360.	36.6	183.000	246.
H DAAMS	GPL	2.00E-05	720.00	0.50	2	36.00	7.200	36.000	54.
H NRT <sup>(5)(6)</sup>	STEL	3.00E-03	1.83	1.00	2	5.49	1.1	5.49	8.25

#### **10.4 Corrective Action for Unacceptable Calibrations**

If an instrument fails to meet the calibration criteria, the instrument will not be used for future analyses and will be tagged or labeled to indicate this status until the problem is corrected. Corrective actions for calibration failures will be implemented and recorded.

A record shall be kept of the calibration of LAB instruments and Class 1 NRT monitors for each method. This record shall include the following information; 1) Date of calibration, 2) instrument/monitor identification, 3) method ID, 4) analyst/operator, 5) calibration standard(s) name, identification number(s) and target concentration(s), 7) instrument/response and/or retention time for each calibration standard tested, 8) results from the regression analysis calculations where applicable, and 9) the results of calibration (e.g., pass, fail). This information will be recorded on AMSCM-OPDC Form 5023, (Appendix C).

#### **10.5 Frequency of Calibration for NRT Monitors**

Calibration of NRT units will be in accordance with (IAW) the instructions given in monitoring SOP MON 33-00-99-05 (including RTAPS from Area 10), which were written with guidance from the manufacturer's operational manual and per this Permit. Calibration criteria are listed below:

1. At a minimum, the calibration will be performed using two injections at the HL with one blank cycle between injections to clear any residual agent (carry over less than 0.2Z).
2. After the calibration has been completed, the instruments will be challenged with an HL challenge.

A successful challenge or calibration before use of an NRT monitor is required if the unit has not received a QP challenge for the intended method during the preceding ten days.

#### **11.0 COLLECTION, HANDLING, CUSTODY AND PRESERVATION**

All actions associated with sample collection, monitoring and generation of data will be accurately documented and will be carried out IAW this document and Army SOPs.

Record keeping begins with standards preparation and continues through Chain of Custody to the Laboratory until the final analytical reports are filed.

Logbooks with pre-numbered pages or permanently affixed computer-generated report forms shall be utilized for record keeping. Numbered pages encourage use of data in sequence and also aid in referencing data through a table of contents ordered according to time, type of analysis, type of sample and /or identity of analyst.

Logbook entries and sample labels shall be completed in ink, accurately. Any corrections necessary to the logbook or sample label shall be made by drawing a single line through the incorrect entry, entering the correct information, and initialing and dating the change.

A unique sample ID number will be affixed to the sample or sample container or the sampling documentation will be detailed enough to be tracked back to that individual sample and provide adequate sample identification.

Complete information shall be entered so that in an examination it can be determined what was done, by whom, when, and what the results were.

Trained personnel IAW this document and site specific SOPs (Appendix B) will perform collection of analytical samples. Stringent sampling methods are employed at the CAMDS Site to ensure the safety of personnel, the environment, equipment, and facilities.

## **11.1 Sample Collection**

The individual collecting the sample shall initiate the transfer of possession and sign the sample collection record. The following types of information, as a minimum, shall be recorded on the sample collection record: 1) unique sample ID, 2) date and time, 3) start and stop flow rates or solid/liquid collection times, 4) start and stop times of the sampling period, 5) weight (gram [g]) or volume (milliliter [mL]) (weight may be determined by personnel performing the analysis.), 6) collection location, 7) operator's name or unique ID number, 8) agent and monitoring level, 9) sample type, and 10) preservatives. Some items may not be applicable to all types of sampling procedures. Deviations from procedures, security, and unusual environmental conditions shall be included in the sample collection record.

### **11.1.1 Class 1 NRT Monitors**

Sample collection for Class 1 monitors is an automated process that operates continuously. Once the monitor has been calibrated and its performance verified, the collection, handling, delivery and analysis of the sample is controlled by the monitoring unit itself. Strip charts and/or data reports generated by these monitors provide evidence of the presence or absence of chemical agent. Strip charts and data reports will identify the location, date, operator or analyst, time and agent being monitored. These charts and reports are quality records and will be maintained accordingly. The Monitoring Division will be responsible for maintaining these records.

The operational log sheet and strip charts will be collected as operations dictate. Strip Charts and reports will be filled out IAW SOP MON 33-00-99-05 (including RTAPS). The supervisor will verify that there is a strip chart or printed report to match each log sheet. After the operational log sheet has been reviewed, the supervisor will then make the copies that will be taken to the data contractor to produce the statistical report. The supervisor will box up the charts and log sheets until arrangements are made to ship to the 40-year storage location.

### **11.1.2 Liquid and Solid Sampling Plan**

Liquid samples are collected from brine tanks and hydraulic oil reservoirs. Examples of solid matrices are residue, salts, and furnace ash. Samples collection must be documented to accurately record the possession and handling for each sample from the moment of collection through the moment of disposal. All transfers of possession must be documented. Transfers of possession will be documented with date, time, and signature of persons receiving and relinquishing possession. Documentation will be maintained up to the time samples are analyzed or destroyed and the accompanying paperwork will be annotated with the sample disposition including date, time, and signatures. The Sample Custodian will check corresponding documentation for completeness and accuracy. The following items must be on each sample collection record:

1. Date and time of collection
2. Collection location
3. Sample type matrix

4. Sampler's number
5. Analyses required
6. Point of contact for results

Any deviation from operating procedures or method parameters, or unusual environmental or operating conditions, will be annotated on the sample collection record.

Solid and liquid samples shall be analyzed IAW specifications in the Waste Analysis Plan (Attachment 2) of this Permit.

#### **11.1.3 Class 1/Historical Methods**

The proper collection and handling of samples (e.g. DAAMS tubes) for Class 1 methods is very important since improper sample collection produces invalid analytical results. All records sampling, and collection, including any change of custody records shall be legible and accurate. Sample identification for DAAMS tubes are provided by the LAB in the form of an ID number that is affixed to the outlet end of each DAAMS tube.

Individuals who collect DAAMS samples to be analyzed at the LAB must document their activities utilizing a Sample Collection Sheet. Pre-printed labels for each DAAMS sample bag shall be corrected if inaccurate at time of sample collection. A straight line shall cross out the mistake, accurate data recorded, and then initialed and dated by the operator. Each collection sheet shall document information on all samples. The following information will be annotated on the collection record.

1. Time aspiration began.
2. Date and time sample ended.
3. Initial sample flow rate.
4. Final sample flow rate.
5. Location of sample collection.
6. Sample collector name and initials.
7. Agent being monitored.
8. Sample type and sample ID number.
9. Miscellaneous visual observations.

#### ***DAAMS Station Logbook***

Individuals, who collect DAAMS samples for analysis, shall record in a logbook (at station location) the initial collection time, station number, aspiration time, and flow rate, for any upset condition such as an alarm or non-routine maintenance (including vacuum pump change out). In addition, change out of the V-G conversion pad shall be documented in the logbook with the time, the date and the operator's initials.

#### **11.2 Receipt of Samples at the Laboratory**

Sample recipients will be trained in the receipt, handling, protection, and retention of samples. The sample recipient shall ensure that all samples are accounted for, undamaged, and properly labeled; damaged samples will be documented on the associated paperwork.

The sample analysis schedule is based on the priority of samples. Lab management and the control room determine priority of sample analysis. Records are maintained that identify the analyst and instrument used to produce sample analysis results. DAAMS samples shall be analyzed within 72 hours of sample collection. Samples results from samples analyzed outside of 72 hours will have those results annotated. Waste screening samples shall be analyzed in accordance with their specific holding time requirements. Samples used for the confirmation of agent have priority over all other samples.

### 11.2.1 Custody Record

An accurate record of the possession and transfer of each sample from the point of its collection will be maintained until it is submitted to the LAB for analysis. The CAMDS Site Monitoring Division will maintain copies of DD Form 1222/locally generated form submitted for analysis pending receipt of the final copy with results. The form will be reviewed prior to placing in storage.

LAB personnel will sign the Chain of Custody tag upon receipt of the samples at the LAB. Samples will be inspected for suspect quality. Any DAAMS tubes that are broken, contain foreign material, or are otherwise abnormal will be documented on the sample paperwork. LAB personnel will inspect sample chain of custody labels to ensure the following information is complete:

<b>Table 11-1:</b>	
<b>Label/Chain of Custody information required for DAAMS tubes.</b>	
<b>DAAMS Tube</b>	<b>Solid/Liquid</b>
Station number	Sample number
Sample flow rates	Sample date and time
Date	Sample location
Sample start and end time	Sample type
DAAMS tube serial number	Method sampled
Signatures of all sample handlers	Signature of handlers
Agent	
AEL	

Analytical and monitoring personnel shall keep an accurate record of the possession and handling of each sample from the moment the sampling device is picked up at the laboratory, by the sample collector, through its collection and transfer back to the laboratory. The person receiving the sample will ensure that the samples are placed in the DAAMS tube or brine receiving areas.

If a person other than the sample collector transports the samples to the LAB, both individuals will initial the sample collection record to indicate that a transfer of possession has occurred.

The DAAMS Sample Receipt Log or the DD Form 1222 shall accompany the samples to the LAB. The following information; 1) time sample began, 2) date and time sample ended, 3) location of sample collection, 4) collector's name or initials, 5) agent being monitored, 6) sample type and 7) miscellaneous visual observations.

Before transferring custody, the sample collector will check to make sure that the sample collection record is complete and accurate with revisions or corrections being made where appropriate. The individual making the correction will initial and date corrections made to the sample collection record. The sample collection records will be filed along with the analytical results.

### **11.3 Sample Storage, Handling and Preservation**

DAAMS sample identification tags are marked to indicate whether they are spiked by indicating if it is a QP or QL sample. Samples will be handled to maintain their integrity. DAAMS sorbent tubes will be capped or sealed in a plastic bag. Once samples are removed from the DAAMS lines and capped they shall remain in the custody of the operator until they can be transported to the LAB for analysis.

The laboratory and monitoring group is responsible for sample storage to ensure that all environment, security, and holding time requirements are met. Samples will either be in the possession of a trained technician or shall be stored in the appropriate secure area.

### **12.0 DAAMS TUBES ANALYSIS**

Samples are aspirated and then analyzed at the WPL, SEL, and GPL hazard levels IAW SOP LAB 66-03-01-01 and SOP LAB 66-00-00-16. The samples are aspirated in duplicate or triplicate sets at the sampling locations and are labeled A, B, or C tubes. The following are the different types of sampling stations.

If agent is detected on the A, B or C tube and a trend is established (3 consecutive sampling periods), at a level below the reporting limit, an investigation of the potential low-level agent contamination will be performed. The Lab shall have developed procedure for tracking low-level agent contamination, within 45 days of the acceptance of this Permit modification, for approval by the Executive Secretary. The DAAMS station will be tracked until the cause of the agent contamination is found or investigation is completed. The sample will be flagged in the DAAMS database, in a manner to allow summary reports to be extracted upon request of the Executive Secretary.

Any Class 2 monitor with a mass detection of agent is considered confirmed agent.

### **12.1 NRT and Co-located DAAMS Stations**

#### **12.1.1 SEL and STEL Stations (NRT Monitor Alarm)**

The STEL samples will be aspirated with the A and B tubes. When the NRT monitor goes into alarm, the A and B tubes will be combined and analyzed on the GC-FPD. If the combined sample analysis result is above the reporting limit, the sample is confirmed. The NRT monitor alarm duration (time) shall be used to determine the concentration of the sample result.

### **12.2 DAAMS-Only Stations**

#### **12.2.1 WPL Stations**

The WPL samples will be aspirated with the A and B tubes. The A tube will be analyzed by GC-FPD. If the A tube analysis result is above the reporting limit (0.5 WPL), the B tube will be

analyzed by a GC-FPD with a dissimilar column. If the B tube analysis result is also above the reporting limit, the sample is confirmed.

#### **12.2.2 GPL Stations**

The GPL samples shall be aspirated with the A, B, and C tubes. The A tube shall be analyzed by a GC-FPD. If the A tube is confirmed, the B tube shall be analyzed with a GC-FPD with a dissimilar column. If the B analysis result is above the reporting limit the sample is confirmed.

The C tube may either be used as a backup for tube A or B, or used to identify the presence of an interferent.

### **13.0 AGENT CONFIRMATION REPORTING**

The confirmation method of analysis must be able to detect the minimum level at which the CAMDS Site is required to confirm and/or report readings. A second method of detection is necessary to provide confirmation of the presence of agent detected by the monitoring system. A method/monitor utilizing a different analytical method from the primary analytical method will be used to confirm the presence of agent. Some methods such as DAAMS require duplicate samples in order to facilitate confirmation.

Any analytical results that are at or above the RL (STEL, SEL, WPL, and GPL), and the confirmation analyses are positive, shall be reported to the Chief, Laboratory Analytical Division, the Control Module Operator (CMO) (documented). The Executive Secretary shall be notified if there are any analytical results that are at or above the RL at the STEL, SEL, or GPL. The report must contain at a minimum the following:

1. Station identification and chemical agent.
2. The date and time of aspiration of the DAAMS tubes; or, if there is an NRT monitor alarm, the time of aspiration shall correspond to the duration of the NRT monitor alarm.
3. The DAAMS sample analytical results reported in terms of mg/m<sup>3</sup>.
4. Instrument type used for confirmation (i.e., GC-FPD, or GC-FPD/MSD).

### **14.0 QUALITY CONTROL**

Quality control provides objective quality evidence that the monitoring system is operating within specified parameters and is capable of detecting and quantifying chemical agent at the required concentrations. To meet this goal, data generated from control samples taken at the CAMDS Site are continuously monitored for day-to-day variations in the routine LAB work and to provide a continuing evaluation of method performance. This information is charted monthly on quality control charts. A sample is defined as the volume of air that is sampled during one automatic cycle of the instrument for the agent and HL being monitored. The following steps describe the way in which this system is implemented.

#### **14.1 Quality Monitoring Control Samples**

A Quality Plant (QP) sample is a sample, which has been spiked with a solution of analyzed dilute chemical agent and exposed to the depot atmosphere. QPs at the HL (1 Z) and RL (0.2 or 0.5 Z) are routinely employed to assess the performance of the NRT monitors, Hewlett Packard

(HP), and DAAMS method. The following information will be recorded regarding QPs challenges: date, monitor ID, agent, operator, QC solution identification type or location (e.g., igloo air/G100, laboratory air/Bldg 130, waste bag/airbag ID, or location of bag, etc.) and found concentration.

## **14.2 Quality Laboratory Control Samples**

### **14.2.1 Quality Control (QC) Samples**

Quality Control Samples demonstrate that the analytical method is within control limits, QC samples are analyzed by the instrument to validate instrument performance. QC samples are spiked with a different set of standards than the set of calibration standards used for calibration. The retention time for all CCV, QLs, and QPs must be within  $\pm 5\%$  of the retention time average for all calibration points on the GC-FPD. The found mass CCV will be  $\pm 15\%$ . The DAAMS QPs must have a found mass of  $\pm 40\%$  of the target mass to be acceptable.

### **14.2.2 Continuous Calibration Verification (CCV) Samples**

The CCV are QC samples spiked by QC LAB personnel at a concentration of chemical agent equal to the concentration of chemical agent at the middle point of the calibration curve. The CCV will function as a monitor on the performance of the analytical instrument. The CCV is spiked with the QC solution. The CCV shall be spiked directly into the DAAMS. As a minimum, the CCV will be performed for every twelve samples analyzed. Evaluation and procedures for the CCV shall be adhered to as required in this Permit.

### **14.2.3 Double Blind Quality Laboratory (QL) Samples**

Double blind QL samples are introduced into the sample batch of environmental samples to function as monitors on the performance of the analytical instruments. The QC specialist on each shift prepares all QL samples. The QLs are DAAMS tubes spiked at random levels between 0.2 and 2.0 AEL and introduced to the analyst as a blind QL into the sample batch. The QL samples are spiked with the QC solution. The QL will function as a monitor on the performance of the analyst and will provide a check on the calibration of the instrument and the standards used to calibrate the instrument. At least one QL shall be prepared for each analyst, each shift, and will be randomly introduced into the DAAMS samples.

The QLs with spike ranges of 0.2 Z to 0.49 Z will be within 50% of the TC. The QLs with spike ranges of 0.50 Z to 1.5 Z will be within 25% of the TC. The QL spike levels at 2.0 Z will yield a positive chemical agent response greater than or equal to 1.5 Z. The QLs will be spiked directly into the DAAMS.

### **14.2.4 Quality Plant (QP) Samples**

The QPs are DAAMS tubes spiked by QC LAB personnel prior to aspiration of the DAAMS tubes in the field. The QP will be spiked at 1.0 Z. The QP will function as a monitor on the overall performance of the field sampling. QPs for each monitoring level will be spiked and aspirated according to Table 8-1. The daily QPs will be analyzed on two dissimilar columns.

## **14.3 Bracketing QC for Historical and Confirmation DAAMS Samples.**

All laboratory instruments (GC-FPD, GC-MSD, etc.) used for analysis as part of a historical or confirmation method shall be bracketed/challenged with an initial calibration verification sample [ICV] and continuing calibration verification samples [CCV]). Class 1 methods will have an ICV

sample immediately following an instrument calibration. The ICV target concentration shall be within the calibration range. All samples will be batched by method, instrument, or calibration range. Each batch will start and end with a CCV and shall include a CCV sample for every 10 samples analyzed. The CCV target concentration shall be equivalent to the monitoring level for each method. All Historical DAAMS and NRT confirmation Samples will be analyzed on a GC configured with a different column than the primary result.

If the percent recovery of the CCV is <85% or > 115%, another CCV will analyzed, if the CCV is within control, then the n analysis is considered valid. Sample results may be qualified when the CCV fails high with the appropriate data qualification. When the second CCV is not within the control limits, but above 50% recovery and all readings on the samples are less than or equal to 50% of the RL, the analysis is valid and the sample is reported as less than the RL. If the CCV is below 50% recovery, the analysis is not valid. Failure of the QC shall require that the secondary tube to be analyzed on an instrument with a different analytical column. Failure of QC for confirmatory DAAMS tubes shall require the primary agent reading to be reported as confirmed.

#### **14.4 DAAMS Tube Spiking**

A calculated volume of analyzed dilute chemical agent is delivered to the DAAMS tube from a micro liter syringe; drawing clean air through the tube for a short time evaporates the solvent. The mass of agent, placed on the tube, is equivalent to that which would be collected if the tube were sampling air contaminated with chemical agent at a concentration equal to either the HL or the RL. After preparation by the LAB personnel, QPs are sent to the monitoring location to aspirate air for the required amount of time and then returned to the lab for analysis.

#### **14.5 NRT Monitors Spiking**

To prepare a spiked sample for an NRT monitor, a known volume of analyzed dilute chemical agent is injected into the sample inlet during the NRT monitor sample cycle. NRT monitors in the VX mode will have injections made through two Silver Fluoride conversion pads. Spikes will be of the correct concentration and volume to deliver the same mass of agent to the instrument as would be collected if the instrument were sampling air-containing agent at the HL or if required at the RL concentration. All NRT monitors shall receive agent challenges and the acceptance criteria will be IAW SOP MON 33-00-99-05.

#### **14.6 QC Sample Loading**

##### **14.6.1 Class 1 NRT Monitors (Fixed and Mobile)**

Fixed site Class 1 NRT monitors that operate, continuously, will be challenged with an HL challenge once a calendar day. There shall be a minimum of 12 hours between daily HL challenges. Fixed site Class 1 monitors that operate intermittently will be challenged with an HL QP before agent operations, and an HL challenge once a calendar day. There shall be a minimum of 12 hours between daily HL challenges.

Fixed site and mobile Class 1 NRT monitors that have been turned off (flame extinguished) for more than eight hours will be challenged with an HL QP before use.

##### **14.6.3 RTAP**

RTAPs utilize NRT monitors with spiked samples prepared as stated in section 14.0. Mobile Class 1 NRT monitors will be challenged with an HL challenge before operations, an HL five hours (if operations are expected to exceed five hours), and an HL at the end of operations each

day. For each NRT monitor, at any time during the operational day, a change of agent requires an HL challenge prior to initiating operations with the new agent.

To verify the sampling system, the NRT monitor can be put in the "Manual Sample" mode and then an HL (1.0 Z) injection made at the end of the distal end of heat-traced sampling lines. The NRT monitor will aspirate for a sufficient period of time, allowing the agent standard to vaporize and travel to the pre-concentrator tube (PCT). The ambient air temperature and the length of the heat-traced sampling line will determine the amount of time required. The reading on the display of the NRT monitor must show  $1.0 Z \pm 25\%$  to be considered within tolerance.

#### **14.7 Class 1 Monitor Performance Charts**

Performance charting monitors variations in the QP challenge results of Class 1 monitors and detects trends in those variations. Performance charts are based on QP data at the HL for each Class 1 monitor. Performance charts, with data for one agent, shall be maintained for each station/monitor in use. The CAMDS Site shall use performance charting for the QP data as described below.

1. Control charts for NRT monitors shall be produced from the flat American Standard Code Information Interchange (ASCII) files provided from the online data collection system. The capability to prepare control charts from manually entered data must be maintained during times that such files are unavailable. In the LAB, control charts should be prepared on a personal computer or microcomputer in a spreadsheet or database software environment.
2. There will be a separate set of control charts for the IDLH, STEL, SEL, and WPL monitoring levels. The NRT monitors require a separate control chart.
3. The performance boundaries for the hazard level charts are defined as follows: the upper performance boundary equals 1.25 Z and the lower performance boundary is equal to 0.75 Z.
4. Daily log sheets require the following information: title of the chart, identification of the agent, identification of each challenge for each given day, station, found concentration, monitor, and the performance boundaries.
5. The performance boundaries for the hazard level charts are defined as follows: the upper performance boundary equals 1.25 Z and the lower performance boundary is equal to 0.75 Z for NRT monitors and + 40 % for DAAMS tubes.

#### **14.8 Control Charts in the Laboratory**

##### **14.8.1 CCV**

After the CCVs are analyzed, the analyst will plot the results on the control chart. The analyst will plot sample results for each calibration level. A separate control chart will be generated for each calibration level. An acceptable CCV found mass will be  $\pm 15\%$ . The TC is the spike level of the CCV. Retention times of the CCV will be  $\pm 5\%$  of the average retention time of all calibration points for the corresponding calibration range of the instrument. A daily CCV report will be required when the daily percent of out-of-control CCV is  $> 20\%$ . The report will be given to the Chief, Laboratory Analytical Division.

#### **14.8.2 QL**

The QL samples shall be plotted on Control Charts monthly, updated, and reviewed. A database will be maintained of all QL samples. The QC Coordinator will review the QC data daily. Corrective action will be documented. Analytical results of QL samples that have concentrations that fall outside of acceptable limits of  $\pm 25\%$  recovery will be accompanied by a written description of the corrective action. Data points may be removed only if proven to be due to human error.

#### **14.8.3 QP**

The QP data will be plotted, updated, and reviewed daily by the QC Coordinator and any corrective action shall be documented when recoveries are less than  $\pm 40\%$  (Appendix E). A separate control chart will be generated for each sample monitoring level. Each chart will contain results data from the previous two weeks. The upper and lower control limits correspond to the upper and lower 40% of the spike concentration. Charts, Corrective Action Reports, and QP data will be submitted to the Laboratory Analytical Division Chief each week. Laboratory Analytical Division will provide copies of charts, Corrective Action Reports, and QP data to Risk Management Directorate. The QP data will be provided to the Statistician every two weeks. If the reporting limit is set higher than 0.5 of an AEL, then QP challenges shall demonstrate a recovery of  $\pm 25\%$ .

#### **14.9 Division QP Review**

Every other Thursday, the Statistician will transmit the QP data results from the previous four weeks to the Chief, Laboratory Analytical Division, and to the Environmental and Monitoring Office (EMO) Project Officer. A Corrective Action Report will be transmitted to the EMO Project Officer no later than six days after a correction is made. The Chief, Laboratory Analytical Division, will review and store all information.

#### **14.10 CCV Out-of-Control Condition**

The CCV will be considered out-of-control when samples spiked with  $\geq 0.3$  ng of dilute chemical agent results are not within  $\pm 15\%$  of the target concentration and retention times exceed  $\pm 5\%$  of the average retention time of the calibration of the instrument.

When two successive CCVs are out-of-control, a calibration standard is analyzed. If the percent recovery is  $\pm 15\%$  of the spike concentration, the LAB QC representative will verify spiking methods and/or solutions. A CCV from a freshly spiked set will then be analyzed. If this CCV is acceptable, the analyses of samples may proceed. If the fresh CCV is out-of-control, the corrective actions are documented, the problem fixed, or the instrument recalibrated. The CMO will be informed when:

1. LAB management makes the determination there is not a sufficient number of instruments available to support the CAMDS Site operation.
2. The LAB is unable to operate due to problems with utilities, or other unforeseen circumstances that dictate the closure of the LAB by the Operations Support Directorate. If the LAB is closed, the Director of Plant Operations, the Director of Risk Management, and the CMO will be notified immediately.

#### **14.11 Control Charts for Monitoring**

The purpose of a monitoring control chart is to monitor the variations in the P&A of routine analysis and to detect trends in those variations. Control charts (based on QP data at the hazard level) will be prepared for all Class I methods. The QP data at the LOQ are not plotted on a control chart. These QP parameters are  $\pm 25\%$  of the TC used to periodically certify the method meets or exceeds minimum standards to detect and quantify chemical agent.

The NRT monitor control charts shall be produced, insofar as possible, from the flat American Standard Code Information Interchange (ASCII) files provided from the online NRT monitor data collection system. The capability to prepare control charts from manually entered data must be maintained during times that such files are unavailable. In the laboratory, control chart preparation should be established on a personal computer or microcomputer in a spreadsheet or database software environment. There will be a separate set of control charts for each family of IDLH, SEL, and STEL. The NRT monitoring systems, ACAMS and MINICAMS<sup>®</sup>, require a separate control charts (e.g., one control chart for all STEL ACAMS and one control chart for STEL MINICAMS<sup>®</sup>). The data collection contractor will prepare the NRT monitor s control charts.

Initial control charts are prepared from the hazard level data collected during the 4-day P&A study performed between method development and implementation. The CERTIFY program provides an estimation of the necessary control and warning limits and the central line for this chart.

#### **15.0 OUT-OF CONTROL INVESTIGATIVE PROCEDURES**

Samples that have been analyzed with the GC-FPD and have been identified as out-of-control will be reanalyzed by using the second or backup DAAMS on a GC-FPD that has been shown to be within calibration control limits.

The QC Coordinator shall investigate the data point in question for an assignable cause and verify that any findings are properly documented. A result may be eliminated, if an assignable cause for the poor result is found to be caused by human error. All other data points shall be plotted.

The Corrective Action Report for an Out-of-Control QP (Appendix E) shall be used for the investigation and the documentation of Out-of Control items.

#### **15.1 LAB Check of Reported Data**

The following items shall be investigated and documented:

1. Check calculations and verify that a proper calibration curve was utilized when calculating QP results.
2. Verify GC set points.
3. Verify the GC is in-control by reviewing the previous QL or CCS.
4. Ensure calibration QP standards are at the same concentration (cross-checking).
5. Verify proper desorption technique was used.
6. Ensure proper sampling at QP station was conducted.
7. Verify flow rates.

8. Ensure proper sampling time was used (check sequencer).
9. Ensure there were no unusual occurrences during aspiration (painting, heating/cooling, etc.).
10. Check for correct/current nitrogen oxides and for silver fluoride conversion pads.
11. Ensure proper QP spiking procedures were used.
12. Verify that QL spikes had good recovery.
13. Verify condition of conversion pads.
14. Verify that correct QP tubes were used.
15. Verify good manifold vacuum.
16. Verify spiking manifold orifices are unclogged.

### **15.2 QP Sampling Spiking Procedures**

Ensure that the proper sampling time is recorded by checking the sequencer. Check for correct/current nitrogen oxides and that the silver fluoride conversion pads are in place. Document that there were no unusual occurrences during aspiration (e.g., painting, heating/cooling, etc.). DAAMS QP samples recoveries shall be within  $\pm 40\%$  or corrective action shall be taken. Verify that the heat-traced lines are operating correctly and that flow rates are within required limits.

Verify the condition of the silver fluoride conversion pads, that QL spikes had good recovery ( $\pm 25\%$ ) that correct QP tubes were used, that the manifold vacuum is sufficient, and that spiking manifold orifices are unclogged.

### **15.3 Out-of-Statistical Control: Class 1 Fixed Methods or Monitors**

The statistician at the CAMDS Site will perform statistical analysis when it is determined that there are out-of statistical control situations. Data will be taken from the daily operational log sheets and input into the statistical report. Bi-weekly reports will be submitted to the Risk Management Directorate, DCD, and CMA for their review.

### **16.0 SAMPLE LINE CHALLENGES AND QC**

To verify that their transmission efficiency remains high, all fixed and mobile sample lines must be challenged prior to their initial deployment, and thereafter once every sixty days  $\pm 5$  days (minimum six times a year). New sample lines shall be tested for interferences prior to introduction of agent. The CAMDS Site shall verify before use that the sample lines and sample probes provide a transmission efficiency of at least 75 percent. CAMDS personnel prior to equipment use will demonstrate this transmission efficiency. Results shall be recorded on the sample line challenge form. All challenge injections shall be made at the distal end of the sample line. If the lines are used for agent VX, the injection will be made onto the silver fluoride pads. Corrective action shall be initiated after two consecutive challenge failures.

A heat traced sample line will be used for air monitoring to aid in the transfer of agent through the sampling system. The data from the sample line challenge and leak test will be documented on the Sample Line Challenge Form (Appendix C) and filed in the Monitoring Division Office. Supervisors shall review all sample line challenge data for accuracy.

Statisticians shall track/trend a continuing baseline study for all line challenge data. If there are two consecutive first challenge line failures then the test frequency shall be increased. Also, V/G conversion pad change outs versus challenges shall be tracked.

Continuing challenges shall demonstrate a recovery of  $\pm 40\%$  of the TC (at the applicable monitoring level) when challenged at the distal end. If alarm set points or RL are set higher than 0.5 of the STEL or WPL, the challenges shall demonstrate a recovery of  $\pm 25\%$ . The challenge will consist of 1.0 Z injection of the agents sampled through that line.

Near Real Time (NRT) monitors shall be used for all sample line challenges where NRT monitors are in proximity to the sample line. This is the preferred method due to immediate knowledge of pass/fail results and will allow corrective action to be taken in a more time efficient manner.

## **16.1 Line Challenges Specific to the MPF Stack**

### **16.1.1 MPF Stack NRT Sample Lines**

MPF NRT sample are challenged through the probe and sample lines, per this Permit every four hours and thus do not need to be in the sample line challenge rotation schedule every 60 days.

### **16.1.2 MPF Stack DAAMS Sample Lines**

Upon approval of this modification the MPF stack DAAMS sample lines for HD and GB shall be challenged once a week for four weeks. If all first sample line challenges pass on these four weekly challenges, then the sample lines shall be challenged again 30 days later. If this 30 day first line challenge passes, then the sample line challenges for the HD and GB DAAMS sample lines, on the MPF stack, shall be challenged once every 60 days  $\pm 5$  days.

DAAMS VX sample lines, on the MPF stack, shall be challenged every four hours when the V-G conversion pads are changed out. All challenges shall demonstrate a recovery of  $\pm 25\%$ . As VX DAAMS sample lines are challenged every four hours they do not need to be in the sample line challenge rotation schedule every 60 days.

## **16.2 NRT Monitors Sample Line Challenge**

If the initial line challenge fails, then the operator shall immediately re-challenge the line. If the second line challenge fails, then corrective action shall be undertaken and all monitoring instrumentation associated with the failed line shall be taken off line. The CMO/EOC shall be notified immediately, that the area is no longer being monitored and the CMO/EOC shall post or restrict the area to personnel without the appropriate PPE until corrective action is completed and the monitoring system is back in control. All notifications and subsequent actions must be documented in the CMO/EOC logbook.

## **16.3 DAAMS Tubes Sample Line Challenge**

Analytical results from the first line challenge using DAAMS tubes shall be available within 72 hours from the time the line was challenged. If the first line challenge fails, then the line shall be immediately re-challenged. The analytical results for the second line challenge shall be available within four hours from the time the line was challenged. If the second line challenge fails, then corrective action shall be undertaken and all monitoring instrumentation associated with the failed line shall be taken off line. The CMO/EOC shall be notified immediately, that the area is no longer being monitored and the CMO/EOC shall post or restrict the area to personnel without the

appropriate PPE until corrective action is completed and the monitoring system is back in control. All notifications and subsequent actions must be documented in the CMO/EOC logbook.

Additionally for perimeter sample line failures, the Emergency Operation Center (EOC) shall be notified that the monitoring station affected is not operational.

#### **16.4 Mobile NRT monitors**

Heated lines for Mobile NRT monitors shall be challenged, at the distal end daily and prior to the beginning of operations, with agent concentrations at the RL, to ensure continuity (IAW Sections 16.0 – 16.2).

In Area 10, a heat traced sample line may be omitted in the Hazardous Waste storage areas, only if the following conditions are met:

1. Agent sample line challenges are injected at the end of the sample line where air enters the sample line at the headwall.
2. During operation the V-G conversion pads shall be changed every hour, and the distal end of the sample line challenged every hour.

#### **17.0 INSTRUMENT ACCEPTANCE TESTING**

Acceptance testing of new analytical and monitoring equipment will be performed prior to field implementation (including sample lines, Probes, airlocks, ventilation filters, DAAMS manifolds). Prior to performing monitoring functions, all sources of possible contamination will be considered and either eliminated or minimized. New sample lines shall be tested for interferences prior to introduction of agent. Verify sample lines and sample probes provide a transmission efficiency of 75 percent. Sorptive tubes, such as DAAMS tubes and transfer tubes, may become contaminated in the manufacturing or shipping process. New batches of tubes must be tested to ensure the tubes are acceptable for chemical agent monitoring. A certified operator will perform acceptance testing. Pre-used analytical or monitoring equipment received from other chemical demilitarization sites or other government agencies are deemed to have already been acceptance tested. The Laboratory Analytical Division will maintain all acceptance-testing records.

#### **17.1 DAAMS Sample and Transfer Tubes**

##### **17.1.1 DAAMS Tubes**

The DAAMS tube fabricator will provide certification, with each lot of tubes supplied, that the tubes conform to the following:

1. The tube sorbent material component is 60/80 mesh, Chromosorb 106 for GB/VX agents and 60/80 mesh, Tenax for GA/H/L agent.
2. Tubes were heated to condition the sorbent overnight at a temperature of 200 °C under a flow of nitrogen.
3. The fabricator will measure the Pressure drop across each tube was measured and met specification requirements.
4. The LAB will verify the vendor's shipping manifest and note this and any anomalies in the Tube Certification Log Book.

### 17.1.2 DAAMS Tube Blanking

DAAMS tubes shall be thermally desorbed, at 200° C, in a heating block for 20 minutes, using a vacuum with a minimum flow rate of 0.5 liters per minute (L/min).

The LAB will analyze a blank chromatogram for all new tubes using a GC calibrated at the low range. If there are no peaks within the chemical agent window, the tube is accepted for chemical certification. If there are peaks within the chemical agent window, the tube is classified as a rejected tube. Tubes will be lab labeled with a unique identification number for the purpose of audit tracking.

### 17.1.3 Chemical Certification

After a lot or batch of new tubes passes the acceptance tests for "blanking", tubes must be chemically challenged to assure that any chemical agent adsorbed can be detected. This is done by randomly selecting tubes in the lot (Table 17.1); spiking them at the appropriate GPL concentration and comparing their recoveries with the average recovery of three identically spiked GPL transfer tubes. Should a tube fail to recover at  $\pm 15\%$  of the GPL, that tube is retested to ascertain if the operator made an error in spiking the tube. A second failure will cause the tube to be rejected. All chemical test data, whether passing or failing, is recorded in the database.

<b>Table 17-1: Sample Sizes for Normal Inspection for a Maximum of Two and One Half Percent Non-Conformance.</b>					
LOT OR BATCH SIZE			SAMPLE SIZE	ACCEPTANCE #	REJECTION #
2	to	8	2	0	1
9	to	15	2	0	1
16	to	25	3	0	1
26	to	50	5	0	1
51	to	90	5	0	1
91	to	150	8	0	1
151	to	280	13	1	2
281	to	500	20	1	2
501	to	1200	32	2	3
1201	to	3200	50	3	4
3201	to	10000	80	5	6
10001	to	35000	125	7	8
35001	to	150000	200	10	11
150001	to	500000	315	14	15
500001	to	OVER	500	21	22

### 17.1.4 Quality Control of DAAMS Tubes

At the CAMDS Site many DAAMS tubes are aspirated, but never analyzed. Unanalyzed tubes are purged and reactivated prior to reuse. Tubes shall be reconditioned at a minimum flow rate of 1000 ml/min and conditioned for 5 minutes at 200 °C.

To ensure the tubes do not contain contaminants or residual chemical agent that would bias future analytical determinations, a percentage (Table 17.1) of the tubes will be pulled from the reprocessed lot and analyzed. The number of tubes to be pulled for analysis is based on ANSI/ASQC Z1.4-1993. The DAAMS tubes that are analyzed by this procedure will be used for SEL sampling.

General Inspection Level I (ANSI/ASQC Z1.4-1993) shall be required with an acceptable Allowable Quality Level (AQL) of 2.5% non-conformance as listed in Table 17-1.

All DAAMS tubes that have been in contact with chemical agent will be analyzed to determine if the tube will produce a blank chromatogram. If the tube does not produce a blank chromatogram, the tube will be decontaminated and discarded as Hazardous Waste.

## **17.2 Silver Fluoride VX Conversion Pads**

A portion of the V-to-G conversion pads also must be sampled to ensure the effectiveness of the conversion of chemical agent VX to methyl ethyl phosphonofluoridate (G-analog). Each new lot of conversion pads will be sampled IAW ANSI/ASQC (Table 17.1). Conversion pads will be randomly selected in the lot, spiked at 1.66 ng chemical agent VX. Their recoveries will be compared with the average recovery of three spikes at 0.88 ng of GB. The peak area for each conversion pad will be divided by 76.5% to account for the conversion efficiency of the pad.

An acceptable pad is one that has a recovery of greater than 75%. A lot will be rejected if the maximum number of rejected pads, as determined by the testing procedure, is obtained. All chemical test data, whether passing or failing, is recorded.

### **17.2.1 Replacement Frequency**

According to NIOSH's recommendation in "*Special Report on VX: Evaluation of Pesticide Interference and Evaluation of Conversion Pad Service Life*," conversion pads for all-level methods will be replaced as listed below for each Class 1 method/monitor station during VX operations. Pad replacement will occur as listed below:

1. For IDLH-levels every eight hours or more frequent at the discretion of the technician.
2. For SEL on the MPF stack the conversion pads shall be changed out every four hours.
3. Conversion pads on the MDC2 ovens shall be changed after every treatment cycle.
4. Filter stacks and midbeds shall be changed every 28 days pads.
5. Other areas at the STEL/WPL and the perimeter DAAMS stations (GPL) shall be changed every 7 days or more frequently at the discretion of the technician.
6. Conversion pads must be stored in a light-tight container to maximize the pad's ability to convert VX to a G analog and to minimize exposure to atmospheric contamination.

## **17.3 NOX Filters**

In order to retain chemical agent Mustard on the DAAMS tube, Nitrogen Oxide pre-filters shall be required for Mustard sample collection. Nitrogen oxides (NOX) pre-filters will be tested by visually inspecting the nitrogen oxides pre-filter for cracks, packing separation, and other physical defects. All Mustard DAAMS sample line challenges shall be completed through the NOX filter.

### **17.3.1 Replacement Frequency**

NOX pre-filters shall be replaced on the MPF stack monitoring instrumentation daily and a minimum of 12 hours is required between pre-filter change-outs. All other Nitrogen Oxide pre-filters, including the Perimeter DAAMS stations, shall be replaced at a minimum of every 14 days.

### **17.4 Pre-concentrator Tubes Replacement Frequency**

Pre-concentrator Tubes (PCT) shall be replaced as needed, by monitoring operators, based upon challenge performance.

## **18.0 DATA REDUCTION, VALIDATION, AND REPORTING**

LAB analysts will record data on the Data Analysis Report and submit it to the QC Coordinator immediately after test results are obtained. The QC Coordinator will immediately review the data for completeness, legibility, accuracy, and any type of unusual problems with the data. The QC Coordinator will initial and date the report upon review completion.

The data will be validated by the QC Coordinator to ensure that CCS were analyzed with every twelve samples, CCS retention times are within 5% of the calibration curve retention time, and the CCV area is within  $\pm 15\%$ .

Checking spike recoveries will validate brines, brine salts, ash, and residue samples. A spiking procedure will be performed on each different matrix type sample and on every tenth sample in an analysis. This matrix spike is not the same as a QP. The term matrix spike and QP are not interchangeable.

DAAMS tube percent recovery analysis shall be tracked. The QC group in the LAB spike sample tubes in the LAB and send them through the entire sampling and analytical process. The results from these tubes are QP data. QP results are plotted on a chart in the LAB and the data are subsequently subjected to a statistical process to determine the recovery rate for a period of one month. These data are then used to apply to the sample analytical results to correct for percent recovery. No correction will be made if the percent of recovery is  $\geq 100\%$ .

In the final step of the validation process, the QC Coordinator will apply the percent recovery to the raw data received from the analysts.

All chemical-agent-positive samples, and the results of any sample designated as a priority sample shall be reported immediately to the CMO/EOC. Any confirmed readings above the reporting limit on samples analyzed in support of DCD shall be reported immediately to the CMO or EOC.

### **18.1 Analytical Results Reporting**

Analytical results will be reported by telephone, through computer lines, or on hard copy government forms. Any test results reported by telephone or computer will be followed with a hard copy report.

DAAMS tube analyses are reported on DD Form 1222, *Request for Analytical Results* or AMSCM-OPDC Form 5025, *Custody Tag*. Brines, brine salts, ash, and residue are reported on AMSCM-OPDC Form 2350, *Analysis Report*.

## **19.0 PREVENTIVE MAINTENANCE**

Preventive maintenance refers to the maintenance that is performed on an instrument at pre-scheduled intervals to prevent or delay the occurrence of a breakdown in the instrument or a component of the instrument. Preventive maintenance will be performed on a regular basis for DAAMS and NRT monitoring instrumentation used in the air-monitoring program. Preventive maintenance schedules will be IAW the instrument's manual or applicable regulation. A supply of parts will be maintained as needed.

### **19.1 DAAMS Stations**

The CAMDS Site DAAMS instrumentation shall be inspected daily for mechanical problems.

At the Perimeter DAAMS stations, the UPS system and heaters shall all so be inspected and results recorded in the station logbook. If any of the perimeter systems is out of control then corrective action must be initiated and the EOC shall be notified that the perimeter monitoring station is not in operation.

### **19.2 NRT Monitors**

The following documentation and verifications are required:

1. Each instrument will have its own log sheet or logbook. This log sheet or logbook will contain the following information:
  2. Serial number, site-specific location, date the instrument was received, and the preventive maintenance schedule.
  3. Instrument maintenance, troubleshooting and repair operations will also be documented in the instrument specific logbook with narrative descriptions, where appropriate, of the initial symptoms of malfunction, the steps taken to locate the problem and the steps taken to rectify the problem.
  4. All routine maintenance functions will be recorded. Each entry will be accompanied by a date, a time of day and the initials of the person who recorded the entry. The instrument logbook will be used to record out of control situations.
1. *Verify ACAMS alarm signal* Notify EOC that test will be done.
  2. Challenge NRT monitors in "Operate" Mode.
  3. Verify that Audible Alarm worked at the NRT and in the EOC

Document results on Net Operational Log

#### **19.2.1 Daily, Weekly and Monthly Maintenance**

The following minimum preventive maintenance on the NRT monitors shall be performed daily, weekly, and monthly.

##### ***Before Daily Chemical Agent Challenge***

1. Verify unit is in the correct agent and concentration mode.
2. Verify unit is in the normal (not calibrated) mode.
3. Before agent challenging, check all flow rates are within  $\pm 10\%$  of the target flow rate.

4. Verify high temperature of Pre-concentrator Tube (PCT) heater.
5. Verify recorder power switch is on.
6. Verify recorder chart switch is on.
7. Verify range for both recorder pens is 10 volts.
8. Perform a zero check on both recorder pens.
9. Verify that alarm lamp, horn, and error lamp come on when the TEST/RESET (RST) push-button switch is pressed.
10. Verify no error message appears on the display.
11. Verify the ZERO/RECORD switch on the recorder is in the record position for both channels.
12. Verify each compressed gas cylinder contains at least 400 pounds per square inch gage (psig). If not, change the cylinder.
13. Check amount of recorder paper remaining. Replenish if necessary.
14. Verify both pens are writing. Replace if necessary.
15. Check ambient temperature. It should be  $30^{\circ} \pm 20^{\circ}\text{C}$ .

***After Chemical Agent Challenge Daily***

1. Verify alarm lamp and horn are operational.
2. Zero checks each pen to ensure they are acceptable.
3. Verify agent peak is centered in the agent gate.
4. Observe the peak shape and baseline to check for gas leaks, electrical malfunction, or column or detector degradation.

***NRT with FPD Weekly***

Check all of the external gas lines and fittings for leaks. Replace NRT PCT, based on agent challenge failures and high carry over, and replace the septum of the agent Standard vials if dry rotted or punctured enough to allow air leak. Verify the equipment is within the following error limits from nominal values, which are as stringent or more stringent than the parameters for the set points listed below.

**ACAMS Parameter Limits:**

- 1) Ambient temperature =  $30 \pm 20^{\circ}\text{C}$ .
- 2) FPD flame temperature =  $30 \pm 20^{\circ}\text{C}$  ( $> 190^{\circ}\text{C}$ ,  $\pm 5^{\circ}\text{C}$  greater than the FPD block temperature).
- 3) FPD block temperature =  $\pm 25^{\circ}\text{C}$ .
- 4) Valve temperature =  $\pm 15^{\circ}\text{C}$ .
- 5) PCT low temperature =  $\pm 30^{\circ}\text{C}$ .
- 6) PCT high temperature =  $\pm 40^{\circ}\text{C}$ .
- 7) Column 1 low temperature =  $\pm 25^{\circ}\text{C}$ .
- 8) Column 1 high temperature =  $\pm 15^{\circ}\text{C}$ .
- 9) Column 2 low temperature =  $\pm 15^{\circ}\text{C}$ .

- 10) Column 2 high temperature =  $\pm 15^{\circ}\text{C}$ .
- 11) Nitrogen =  $\pm 3$  mLs of the set point
- 12) Hydrogen =  $\pm 3$  mLs of the set point
- 13) Air =  $\pm 3$  mLs of the set point
- 14) Sample =  $\pm 20\%$  of flow rate

MINICAMS Parameter Limits:

- 1) Ambient temperature =  $30 \pm 20^{\circ}\text{C}$ .
- 2) FPD flame temperature =  $\pm 99^{\circ}\text{C}$
- 3) FPD block temperature =  $\pm 25^{\circ}\text{C}$ .
- 4) Inlet temperature =  $\pm 15^{\circ}\text{C}$ .
- 5) PCT low temperature =  $\pm 30^{\circ}\text{C}$ .
- 6) PCT high temperature =  $\pm 40^{\circ}\text{C}$ .
- 7) Column low temperature =  $\pm 25^{\circ}\text{C}$ .
- 8) Column high temperature =  $\pm 30^{\circ}\text{C}$ .
- 9) Nitrogen =  $\pm 5$  psi of the set point
- 10) Hydrogen =  $\pm 5$  psi of the set point
- 11) Air =  $\pm 5$  psi of the set point
- 12) Sample =  $\pm 20\%$  of flow rate

***Consecutive Calibration Failures***

If a NRT monitors fails three consecutive calibrations then the NRT monitor shall be replaced.

**19.3 Preventive Maintenance Program CAMDS LAB Only**

A preventive maintenance schedule will be established for each instrument utilized in the analysis of samples covered in this QC Plan. This schedule will contain a list of preventive maintenance requirements and the frequency by which those requirements will be performed. No instrument will be used until its daily preventive maintenance checklist has been completed and the preventive maintenance logbook checklist updated. Examples of the preventive maintenance check lists are provided in Appendix D.

If an instrument does not operate within specified parameters, the Lead is notified, the problem is investigated, the problem is corrected, or the instrument is replaced. The action taken is documented.

**19.4 Out-of-Control Situations**

When a Class 1 monitor system is in an out of control situation, (two successive QP challenges that exceed  $\pm 25\%$  of the target concentration on a given day) immediate corrective action will be taken. The following information will be recorded on an operational log sheet regarding each out of control situation:

1. What conditions prompted the out of control situation.
2. Who was notified of the problem.
3. What corrective action procedures were implemented.
4. Who performed the corrective action.
5. How long the instrument was out of control.
6. How long an instrument was out of service due to the corrective action procedure.
7. What was the root cause of the problem.

## **19.5 Preventive Action**

The goal of the preventive action is to eliminate the cause of potential non-conformity to the quality system. The identification of causes of potential non-conformity to the quality system will be performed during internal and external audits, monthly reviews of agent challenge performance charts, and management review.

## **19.6 Systemic Problems**

All of the CAMDS Site personnel are required to report systemic problems to their immediate supervisor. The supervisor will assign personnel to investigate the problem, formulate corrective actions, implement the corrective actions and document their action in a memorandum. The supervisor will be responsible for ensuring that these corrective actions have effectively eliminated the problem. Documentation of follow-up actions taken by the supervisor will be recorded on a memorandum.

## **20.0 QUALITY CONTROL SUMMARY REPORTS**

The Quality Assurance Control (QAC) personnel shall review, date and initial performance charts, strip charts and monitoring data (sample size to be determined by the QAC) for correctness prior to release for storage as quality records. Non-conformance will be annotated on the proper documents (i.e., memorandum, CAR, etc.).

The QAC shall generate a proficiency report monthly. Monitoring shall review the report, and implement (with documentation) any required corrective action.

Monitoring shall submit all operational log sheets to the statistician and all pertinent challenge results will be manually entered into a database. An INACCMO report will be generated; producing calculations of accuracy of all challenges and records. The INACCMO report will be distributed to the Chief of Monitoring and Director of Operations Support. After data is reviewed by DCD the report is forwarded to CMA for review and approval. Data that does not pass an in-process or final inspection shall be considered non-conforming and corrective action shall be initiated. The Monitoring division will maintain the records.

## **21.0 EXTERNAL/INTERNAL AUDITS**

### **21.1 External Audits**

External audits either announced or unannounced, will be conducted by CMA on an annual basis. Additional periodic audits will be conducted by a qualified organization as requested by the CMA. All documents and data produced by DCDs chemical monitoring personnel are subject to

audit. During an audit, the CMA will brief the Commander or designated representative on any findings and provide written documentation of them.

## **21.2 Internal Audits**

The Operations Support Directorate shall conduct internal audits on a monthly basis, at a minimum. An audit checklist, approved by the appropriate management representative, and will be used to conduct the audit. In addition, any personnel contacted during the audit will be recorded on the checklist.

Monthly audits will include, but is not limited to, review of any quality records such as strip charts, trend charts, operator certification records, maintenance records for monitoring equipment, certification records of monitoring equipment, and operator proficiency.

## **21.3 Audit Reporting and Corrective Action**

Deficiencies from an internal audit, management review, or external audit will be documented. The representative from the organization conducting the audit will distribute the memorandum to personnel having direct responsibility for the area where the deficiency occurred. The responsible personnel will ensure that an investigation is performed to determine the cause of the deficiency, corrective actions are developed and documentation of how these corrective actions were implemented is recorded in a memorandum. Once the corrective actions have been implemented, a memorandum is returned to the organization that performed the audit to close out any findings.

## **22.0 DOCUMENTATION**

Information pertaining to conditions that may affect the results of chemical monitoring samples must be documented. Documentation is necessary to validate that operations are accurately performed IAW regulations and written procedures. Records will be legible, accurate, generated using permanent ink, and maintained to support and substantiate all quality related activities. The CAMDS Site Monitoring Division shall maintain all quality records.

### **22.1 Document and Data Control**

Quality documents such as Quality Manual, QC Plans, Quality Plans, SOPs and stand-alone forms will be identified by title.

### **22.2 CLMQCP Approval and Issue**

The CLMQCP is a controlled document and distribution will be as specified on the document distribution list. Changes and revisions will be published and distributed as necessary to keep the Plan current. At a minimum, a current copy of the CLMQCP shall be maintained at every laboratory and monitoring right-to-know center at the CAMDS Site.

### **22.3 Document Changes/Modifications**

All changes to this Permit require approval by the Executive Secretary.

Changes to any non-Permit documents will be reviewed and approved by the same organizations that perform the original review and approval unless specifically designated otherwise. The document control system will ensure that documents are reviewed and updated as necessary. The designated organization will have access to pertinent background information upon which to base

their review and approval. Where practicable, the nature of the change will be identified in the document or the appropriate attachments. Obsolete documents will be promptly removed from all points of issue or activity locations or otherwise marked to preclude unintended use.

#### **22.4 Standing Operating Procedures (SOP)**

Appendix B of this LMQCP lists the monitoring and LAB operation SOPs that are incorporated by reference into this Permit. Any changes to these SOPs require approval by the Executive Secretary unless equivalent or superior equipment, materials, methods or procedures are used which do not effect the quality of the data or involve a decrease in documentation. The Permittee shall place in the operating record (prior to the institution of such revision) the revision, accompanied by a narrative explanation, and the date the revision became effective. The Executive Secretary may judge the soundness of the revision during inspections of the CAMDS Site and take appropriate action.

Current versions of the SOPs shall be made available to all operators. A controlled copy of the SOPs, in Appendix B, shall be provided, within forty-five days of this Permit modification, to the Executive Secretary.

#### **22.5 Analytical Data.**

Analytical results are maintained in an electronic database and shall be available for review as necessary.

#### **22.6 Control of Quality Records**

All quality records must be legible and identifiable, recorded in permanent ink or on computer storage media stored so they are readily retrievable. Computer-stored data is printed out daily and archived. The Executive Secretary may review any monitoring or laboratory form and require the form to be updated with additional compliance documentation.

Samples of the CAMDS Site monitoring NRT monitor operational daily log, precision and accuracy data form, and Sample line challenge form are provided in Appendix C. The following form revisions are not subject to the requirements of this Permit and may be revised at the Permittee's discretion:

1. The formatting of forms
2. Adding supplementary information

## **Attachment 3**

### **Appendix A**

#### **Personnel Responsibilities**

## **1.0 Personnel Responsibilities**

Program Manager for the Elimination of Chemical Weapons (PMECW) has directed that a Site-specific LMQCP be established and considered part of the overall quality program at each demilitarization site.

### **1.1 Director of Operations Support**

The Director of Operations Support shall coordinate interactions between CMA, DCD management and Laboratory and Monitoring Divisions. Duties shall encompass the following:

1. Direct the overall activities of the Operational Support Directorate.
2. Request shipment of CASARM standards through the PMECW CASARM Project Officer.
3. Review and approves all SOPs and Permit controlled documentation.
4. Advises the base Commander of any issues or problems.

### **1.2 Chief, Laboratory Analytical Division**

The Chief, Laboratory Analytical Division will:

1. Direct the overall activities of the LAB Support Division.
2. Review control charts to ensure the LAB remains within control parameters.
3. Notify Director of Operation Support when shipments of CASARM standards are required.
4. Advise Director of Operation Support of any problems or improvements in the QC Plan.
5. Ensure that LAB Support Division personnel are trained to perform LAB functions safely, with precision and accuracy. Ensure training complies with the Chemical Hygiene Plan.
6. Oversee the purchase of new instruments and materials.
7. Ensure that all equipment is calibrated as required by the Site Calibration Plan.
8. Sign SOPs to authorize work procedures.

### **1.4 Laboratory Analytical Division (Government) Quality Control Leader**

1. Ensure Stock A standards preparation from CASARM is performed IAW CLMQCP guidelines.
2. Ensure analytical procedures have proper QC measures applied to generate sound, defensible data. When out-of-control conditions are experienced the QC Leader will conduct an investigation to determine the cause for the out-of-control condition and take corrective actions. The QC Leader will notify the Monitoring Division of the out-of-control condition to enable the Monitoring Division to make investigations for corrective action. The QC Leader will submit test results IAW the Data Management Plan contained in this document.
3. Verify that Preventive Maintenance on instruments is up-to-date and documented.

**1.5 Laboratory Analytical Contractor Chief, Analytical Group.**

1. Ensure that contractor LAB Support Division personnel are trained to perform LAB functions safely, with precision and accuracy. Ensure training complies with the Chemical Hygiene Plan.
2. Ensure compliance to CLMQAP, SOPs, and QC Plan.
3. Direct QC activities of the LAB to conform to CLMQCP requirements.
4. Ensure LAB equipment is properly calibrated before samples are analyzed.
5. Review analytical requirements to ensure analyses are made in a timely and accurate manner.
6. Ensure the LAB control status is known and samples have been analyzed and reviewed on a priority basis. Ensure priority samples are designated and receive expedited analysis.
7. Ensure out-of-control conditions are addressed as required by the CLMQCP.
8. Ensure that any chemical alarms that have been confirmed by GC analysis have been addressed IAW the proper analytical and QC procedure.
9. Observe LAB operations for safe, efficient, orderly conduct.

**1.6 Laboratory Analytical Contractor Quality Control Leader.**

1. Ensure sufficient numbers of CCV, QL, and QPs are prepared. Maintain accurate records of the preparation and results of CCV, QL, and QPs.
2. Calculate analytical results of all hazard level QPs. Compare with acceptable limits and notify appropriate personnel when QPs are out-of-control. Document all findings. Conduct investigations when QP results fail to meet acceptable criteria. Document investigation findings for out-of-control conditions. Submit QC data, such as QP charts. Report to Chief, Laboratory Analytical Division as requested.
3. Report to Director of Operations Support.

**1.7 Government and Support Contractor Chemists and Technicians.**

1. Perform QC functions to ensure the analytical results produced in the LAB will accurately and precisely reflect the status of the CAMDS Site Air Monitoring System.
2. These functions will ensure the system is in-control, or, if the system is out-of-control, that immediate and effective corrective actions are initiated.
3. Maintain Preventive Maintenance Records for the GCs used for sample analysis. Maintain records of daily maintenance performed.

**1.8 Chief, Monitoring Division**

1. Direct the overall activities of the Monitoring Division.
2. Implement and manage QC procedures and internal audits.
3. Ensure monitoring procedures are accurate and performed in a timely manner.
4. Complete all baseline studies on schedule and ensure all data are submitted to CMA Laboratory officers.

5. Schedule work and assignments in a productive, accurate, timely, and safe manner.
6. Ensure preventative maintenance is applied in a systematic and timely manner.

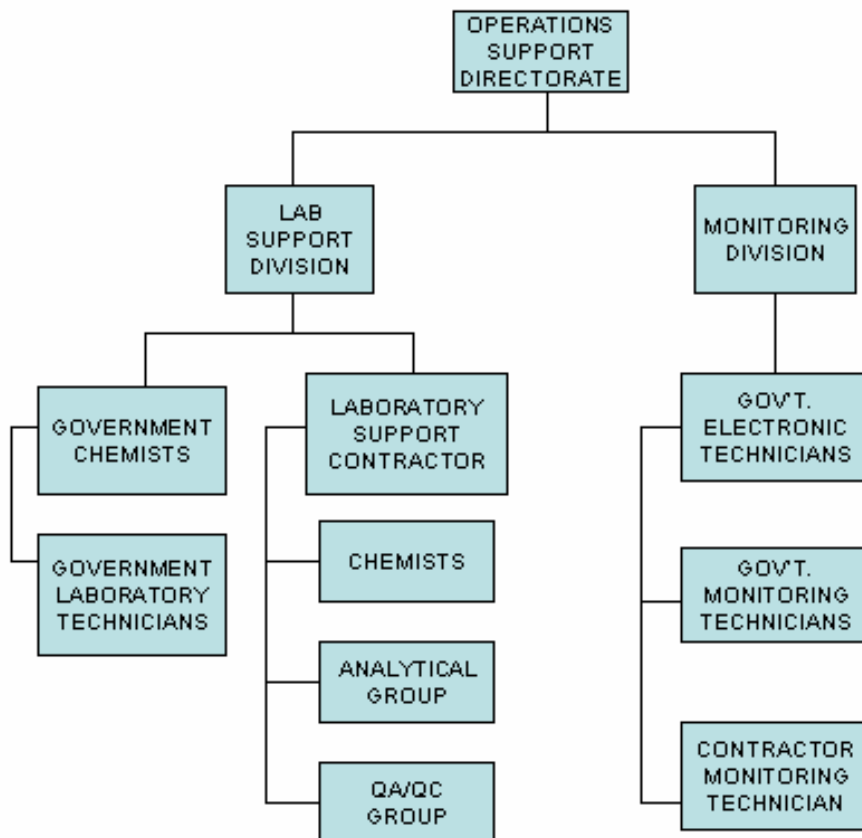
**1.9 Leaders, Monitoring Division**

1. Ensure sampling stations are functioning properly (agent challenges, prefilters, conversion pads, PCTs, heat trace, etc.).
2. Ensure all samples are delivered in a timely manner to the LAB for analysis.
3. Perform daily QC inspections.

**1.10 Operators, Monitoring System**

1. Challenge and calibrate NRT monitors.
2. Collect DAAMS samples.
3. Fill out custody sheets.
4. Perform maintenance and preventive maintenance on monitoring equipment and note time and date for each procedure.
5. Record information in logbooks.
6. Keep all sampling locations, sample equipment, and the monitoring shop clean, safe, and efficient.
7. Comply with SOPs, regulations, and instructions.
8. Record all alarms and malfunctions, and corrective actions taken.
9. Record all agent challenges and quantities of dilute agent standards used.
10. Properly dispose of hazardous wastes.

### OPERATIONS SUPPORT DIRECTORATE ORGANIZATION CHART



CAMDS  
LMQP  
April 2008

## **Attachment 3**

### **APPENDIX B**

**The CAMDS Site Part B Permit Controlled CLMQCP SOP's**

**THE CAMDS PART B PERMIT CONTROLLED CLMQCP SOP'S**

<b>Document No.</b>	<b>Description/Document Title</b>	<b>Rev. No.</b>	<b>Rev. Date</b>	<b>DSHW Approval Date</b>
LAB 66-00-00-09	Analysis for Organic Impurities by GC/MSD	Change 2	12/18/02	5/1/03
LAB 66-00-00-10	GB Purity Determination	Rev. 1	11/25/03	5/21/04
LAB 66-00-00-11	HD Purity Determination	Rev. 1	11/25/03	5/21/04
LAB 66-00-00-12	VX Purity Determination	Rev. 1	11/25/03	5/21/04
LAB 66-00-00-16	Analysis of VX and GB DAAMS/NRT Monitor Tubes	Rev 5	3/11/08	8/12/08
LAB 66-00- 00-19	Extraction of VX, GB, and HD from Demilitarization protective Ensemble (DPE) and Polyvinyl Chloride (PVC) Film	Rev. 1 Chg 2	9/12/05	6/26/06
LAB 66-00-00-20	Analysis of Waste for GB, HD, and VX by Gas Chromatograph/Mass Selective Detector	Rev 5	3/12/08	8/12/08
LAB 66-00-00-22	DAAMS Agent (GB, HD, and VX as G-Analog) Analysis using a GC with a FPD and MSD	Rev 3	3/12/08	8/12/08
LAB 66-00-01-06	Preparation of Dilute Agents from CASARM	Rev 5	3/12/08	8/12/08
LAB 66-00-02-02	Analysis of Liquid or Residue by High Performance Liquid Chromatography (HPLC) for TNT, Tetryl, RDX, and Nitroglycerine	Rev. 1	4/13/04	5/21/04
LAB 66-03-01-01	Analysis of HD DAAMS/NRT Monitor Tubes	INACTIVE SOP		
LAB 66-03-01-02	Analysis of HD Liquid Waste, Brine Salts, Residue, and Furnace Ash Samples	Rev. 3	3/13/07	2/19/08
LAB 66-10-00-01	GB Analysis by Gas Chromatograph/Mass Selective Detector	New	12/11/00	12/10/01
LAB 66-10-04-01	Analysis of GB Liquid Waste, Brine Salts, Residue, and Furnace Ash Samples	Rev. 3	3/30/07	2/19/08
LAB 66-20-00-01	VX Analysis by Gas Chromatograph/Mass Selective Detector	New	12/11/00	12/10/01
LAB 66-20-00-02	Determination of EA 2192 in PAS Brine and SDS by HPLC/MS	Rev 4	3/12/08	8/12/08
LAB 66-20-00-03	Determination of EA 4196 in PAS Brine and SDS by GC/MSD	Rev. 2	3/30/07	2/19/08
LAB 66-20-01-01	Analysis of VX Liquid Waste, Brine Salts, Residue, and Furnace Ash Samples	Rev 3	3/12/08	8/12/08
LAB 66-30-00-01	HD Analysis by Gas Chromatograph/Mass Selective Detector	New	12/11/00	12/10/01

Document No.	Description/Document Title	Rev. No.	Rev. Date	DSHW Approval Date
LAB 66-70-01-01	Analysis for GA Depot Area Air Monitoring System (DAAMS) Tubes	Rev. 1 Chg. 3	02/01/99	Original Permit (9/24/99)
LAB 66-70-01-02	Analysis of GA Brine, Ash, and Residue	New	02/01/99	Original Permit (9/24/99)
LAB 66-80-99-02	Lewisite Purity Determination	Chg 3	12/16/02	5/1/03
LAB 66-80-99-03	Liquid Lewisite Analysis by HPLC with MS	NEW	9/24/01	5/1/03
MON 33-00-99-05	NRT Chemical Agent Challenge Procedures	Rev 12	5/13/08	8/12/08
MON 33-00-99-08	Teflon Sample Line Challenge	Rev 10	5/13/08	8/12/08
TT-0000-M-147	Storage and Accountability of RDTE Standard Solutions	Rev 4 Chg 1	11/08/08	11/25/08

## **Attachment 3**

### **Appendix C**

#### **Monitoring Forms**

Reviewed by:		NRT Sta. No. <u>555</u>						
Mon. Rep. _____		S/N: <u>88-02-130</u>						
QC Rep. _____		Agent/Z level <u>1</u>						
NRT OPERATIONAL LOG (10.0 Z A/C)								
DATE		8-12	8-13	8-14	8-15	8-16	8-17	8-18
TIME	Offline	1708	1623	1618	1605	1922	1829	1712
	Online	1723	1636	1631	1619	2259	1842	1728
AGENT		4216-17	4216-24	4216-24	4216-24	4216-17	4216-17	4216-186
OPERATOR INITIALS								
HAZ CHALLENGE		.94	1.03	1.00	.90	.80	1.06	1.07
	.97 P	N/A	N/A	N/A	N/A	N/A	N/A	N/A
CARRYOVER		.11	.12	.13	.13	.08	.07	.08
LOQ		.20	.19	.21	.18	.23	.25	.24
RETENTION		86	86	87	86	86	86	87
CAL H*		21.3	21.3	21.3	21.3	21.3	21.3	21.3
AVG SAMPLE FLOW		731	708	703	670	677	651	642
DESSICANT FILTER		OK	OK	OK	OK	OK	OK	OK
AMBIENT TEMP		33	34	35	34	33	32	32
N2 (Tank Regulated)		800	800	775	700	700	690	660
H2 (Tank Regulated)		1600	1500	1400	1300	1120	1010	940
AIR (Tank Regulated)		A/B	A/C	A/C	A/C	A/C	A/C	A/C
NRT Horn Sounds		Yes	Yes	Yes	Yes	Yes	Yes	Yes
M42 Horn Sounds		N/A	N/A	N/A	N/A	N/A	N/A	N/A
10.0 Z CHALLENGE		9.41						
NRT Changeout	Date	Ser. No.		Date	Ser. No.			
Comments								
8-16-04 Passed A/C at .80 N/A PM New POT A/C at 1.04 N/A A/C								
PMT VOLTAGE <u>675</u> OPER INIT.								

AMSCM-OPDC Form 5023-1-R-E, 28 Aug 02, REV, 21 Aug 04

Proponent: Director of Operations Support

Precision & Accuracy Data Form

INSTRUMENT: ☒ NRT ☐ GC for GC column type: DB-1  
INSTRUMENT #1 SER. NO.: 3351 LOCATION: MPF Stack  
INSTRUMENT #2 SER. NO.: \_\_\_\_\_ LOCATION: \_\_\_\_\_  
AGENT: VX VIAL NUMBER: A-F CONCENTRATION: UNKNOWN  
AGENT: \_\_\_\_\_ VIAL NUMBER: \_\_\_\_\_ CONCENTRATION: \_\_\_\_\_

Parameter Set Points

1. Sample Flow Rate 1000  
2. Retention Time 81  
3. Sample Time 70-300  
4. Voltage 650  
5. CAL H 830

Target Concentrations

.00, .5, .75, 1.0, 1.5, 2.0

ANALYST/OPERATOR: Name

Injection #	1	2	3	4	5	6	7	8	9	10	11	12
Agent	VX	VX	VX	VX	VX	VX	VX	VX	VX	VX	VX	VX
Injection Vol.	5ul	5ul	5ul	5ul	5ul	5ul	5ul	5ul	5ul	5ul	5ul	5ul
Vial ID	A	B	C	D	E	F	F	E	D	C	B	A
Recovery Z	0.0	.49	.75	1.01	1.55	2.01	1.99	1.56	.99	.74	.51	0.0

Start Time 0800 End Time 1100 Instrument # 3351

Injection #	1	2	3	4	5	6	7	8	9	10	11	12
Agent												
Injection Vol.												
Vial ID												
Recovery Z												

Start Time \_\_\_\_\_ End Time \_\_\_\_\_ Instrument # \_\_\_\_\_

Injection #	1	2	3	4	5	6	7	8	9	10	11	12
Agent												
Injection Vol.												
Vial ID												
Recovery Z												

Start Time \_\_\_\_\_ End Time \_\_\_\_\_ Instrument # \_\_\_\_\_

Injection #	1	2	3	4	5	6	7	8	9	10	11	12
Agent												
Injection Vol.												
Vial ID												
Recovery Z												

Start Time \_\_\_\_\_ End Time \_\_\_\_\_ Instrument # \_\_\_\_\_

### TEFLON SAMPLE LINE TEST DATA SHEET

Station No.		Month Scheduled		Date Performed	
Agent: (circle one) GA HD VX GB L		Leak Test PASS / FAIL		Restriction PASS / FAIL	
Agent Challenge Method (circle one)		Length of Sample line in Feet (approx)		Heat Trace Operational	
ACAMS MiniCAMS DAAMS		YES NO		Sample Point Verified YES NO	
ACAMS S/N:		Aspiration Time		Hours Minutes Seconds	
Standard S/N:		Std Strength ng/mL		Amount Injected ng/mL	
ACAMS FORMULA					
SAMPLE LINE A/C RESULTS (Range = .60 to 1.40 Z) _____					
ACAMS A/C RESULTS (Range = .75 to 1.25 Z) _____					
Sample DAAMS S/N: _____					
Agent Std. Conc. _____ ng/μL		Agent Std. S/N _____			
Quantity Injected _____ μL		Aspiration Time: _____ Hours Minutes			
DAAMS FORMULA					
SAMPLE LINE DAAMS RESULTS = $\left( \frac{\text{QTY Injected} \times \text{Standard Concentration}}{\text{QTY Injected} \times \text{Standard Concentration}} \right) \times 100 = \text{_____} \% \text{ Recovery (7.40\%)}$					
Analysis Method: GC ACAMS (Circle One)					

Challenger's Signature: \_\_\_\_\_

DAAMS Set #1 Agent \_\_\_\_\_  
12 Hour DAAMS START TIME: \_\_\_\_\_ STOP TIME: \_\_\_\_\_

TEMP. DAAMS TUBES #1 #2 START TIME: \_\_\_\_\_ STOP TIME: \_\_\_\_\_

12 Hour DAAMS START TIME: \_\_\_\_\_ STOP TIME: \_\_\_\_\_

DAAMS Set #2 Agent \_\_\_\_\_  
12 Hour DAAMS START TIME: \_\_\_\_\_ STOP TIME: \_\_\_\_\_

TEMP. DAAMS TUBES #1 #2 START TIME: \_\_\_\_\_ STOP TIME: \_\_\_\_\_

12 Hour DAAMS START TIME: \_\_\_\_\_ STOP TIME: \_\_\_\_\_

Supervisor or QC Rep. Signature: \_\_\_\_\_ Date: \_\_\_\_\_

Comments: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

## **Attachment 3**

### **APPENDIX D**

#### **Laboratory Sample Maintenance Checklists**

## DAILY PREVENTIVE MAINTENANCE CHECKLIST

Date: 1 Dec. 04 GC Number: 1  
Night Analyst: (Y) Day Analyst: CEO

Calibration(s) Performed During Shift:

Night: None Day: NONE

Instrument Gases Checked Today.....(N) ✓ (D) ✓

### GC PARAMETERS:

Signal.....(N) 16.2 (D) 18.4  
Oven Temp.....(N) 65° (D) 65°

### BACK INLET:

Inlet Purge Time.....(N) 1.00 (D) 1.00  
Inlet Pressure (Prep Run Mode). (N) 14.88 (D) 15.04  
Inlet Flow (Prep Run Mode).....(N) 80.6 (D) 80.5

### FRONT INLET:

Inlet Pressure.....(N) 7.98 (D) 7.98  
Inlet Flow.....(N) 12.9 (D) 17.0

### HYDROGEN GENERATOR:

Pressure.....(N) NA (D) NA  
Water Level (Fill if less than half) (N) ↓ (D) ↓

### DESORPTION BLOCKS:

Temperature.....(N) ✓ (D) ✓  
Flow-1L/min through DAAMS Tube (N) ✓ (D) ✓

### MISCELLANEOUS:

Area Cleaned.....(N) ✓ (D) ✓

### MAINTENANCE PERFORMED:

## CHECKLIST D-1. MONTHLY MAINTENANCE SCHEDULE

### Monthly Maintenance Schedule

GC Number 123456789XX

DATE	DATE PERFORMED	INITIALS	ANNOTATED IN LOGBOOK
Jan	1-8-04	HA	yes
Feb	2-15-04	HA	yes
Mar	3-12-04	HA	yes
Apr			
May			
Jun			
Jul			
Aug			
Sep			
Oct			
Nov			
Dec			

### Semi-Annual Maintenance Schedule

DATE	DATE PERFORMED	INITIALS	ANNOTATED IN LOGBOOK
Jan			
Jul			
Jan			

AMSCM-OPDC Form 5044-R-E, 11 Feb 04

## **Attachment 3**

### **Appendix E**

#### **Corrective Action Report Sheet for Out-of Control QPs**

**Figure E-1. AMSCM-OPDC Form 5039-R-E, Corrective Action Report for an Out-of-Control QP Datasheet.**

<b>CORRECTIVE ACTION REPORT FOR AN OUT-OF-CONTROL QP</b>																	
Generator:	<b>USACAMDS Activity, Lab Spt Div, Analysis Branch</b>																
CAR #:	GC #:																
Station ID:	Agent:																
Aspiration Date:	DAAMS Tube #:																
Aspiration Time:	ng Recovered:																
Scenario:	% Recovery:																
Analytical Date:	Curve Type:																
<b>A. Analysis Branch Check List</b>																	
1. Analytical Procedures																	
<input type="checkbox"/>	a. Check spreadsheet calculations and verify that proper GC and calibration curve was utilized when calculating QP results.																
<input type="checkbox"/>	b. Verify the chromatogram. Check for integration errors, interferences, and baseline shift.																
<input type="checkbox"/>	c. Verify the GC is in control by reviewing the previous Cal Chk result. Cal Chk recovery = _____.																
<input type="checkbox"/>	d. Check for sufficient manifold vacuum. Check for clogged orifices and correct temperature on the desorption manifold.																
<input type="checkbox"/>	e. Verify that proper desorption technique was used.																
<input type="checkbox"/>	f. Check to see if QL (in-house) spike had good recovery. QL recovery = _____.																
<table style="width: 100%; border: none;"> <tr> <td style="width: 50%;">Is there an assignable cause for the above mentioned QP?</td> <td style="width: 10%; text-align: center;"><b>Yes / No</b></td> <td style="width: 40%;"><b>(circle one)</b></td> </tr> <tr> <td>Spiking Error</td> <td>Severe Weather</td> <td>Desorption Malfunction</td> </tr> <tr> <td>Integration Error</td> <td>Broken DAAMS Tube</td> <td>Operator Error</td> </tr> <tr> <td>Power Failure</td> <td>Wet Tube</td> <td>Other:</td> </tr> <tr> <td>GC Malfunction</td> <td>Broken Transfer Tube</td> <td><b>(circle any that apply)</b></td> </tr> </table>			Is there an assignable cause for the above mentioned QP?	<b>Yes / No</b>	<b>(circle one)</b>	Spiking Error	Severe Weather	Desorption Malfunction	Integration Error	Broken DAAMS Tube	Operator Error	Power Failure	Wet Tube	Other:	GC Malfunction	Broken Transfer Tube	<b>(circle any that apply)</b>
Is there an assignable cause for the above mentioned QP?	<b>Yes / No</b>	<b>(circle one)</b>															
Spiking Error	Severe Weather	Desorption Malfunction															
Integration Error	Broken DAAMS Tube	Operator Error															
Power Failure	Wet Tube	Other:															
GC Malfunction	Broken Transfer Tube	<b>(circle any that apply)</b>															
If an assignable cause has been found, briefly explain what was done to correct the problem.																	
<table style="width: 100%; border: none;"> <tr> <td style="width: 40%;">QA/QC Signature:</td> <td style="width: 60%;">Date:</td> </tr> </table>			QA/QC Signature:	Date:													
QA/QC Signature:	Date:																
<b>B. Monitoring Branch Check List</b>																	
1. Monitoring Procedures																	
<input type="checkbox"/>	a. Verify flow rates.																
<input type="checkbox"/>	b. Ensure proper sampling time. Check sequencer.																
<input type="checkbox"/>	c. Ensure no unusual occurrences(engineering or environmental).																
<input type="checkbox"/>	d. Check for correct/current NOx/conversion pad usage.																
<input type="checkbox"/>	e. Verify heat trace for sample line is on.																
<table style="width: 100%; border: none;"> <tr> <td style="width: 50%;">Is there an assignable cause for the above mentioned QP?</td> <td style="width: 10%; text-align: center;"><b>Yes / No</b></td> <td style="width: 40%;"><b>(circle one)</b></td> </tr> </table>			Is there an assignable cause for the above mentioned QP?	<b>Yes / No</b>	<b>(circle one)</b>												
Is there an assignable cause for the above mentioned QP?	<b>Yes / No</b>	<b>(circle one)</b>															
If an assignable cause has been found, briefly explain what was done to correct the problem.																	
<table style="width: 100%; border: none;"> <tr> <td style="width: 40%;">Operator Signature:</td> <td style="width: 60%;">Date/Time:</td> </tr> </table>			Operator Signature:	Date/Time:													
Operator Signature:	Date/Time:																

## **Attachment 3**

### **Appendix F**

#### **Individual Certification Sheet (AMSCM-OPDC Form 5043-R-E)**

**AMSCM-OPDC Form 5043-R-E, Individual Certification Sheet.**

**INDIVIDUAL CERTIFICATION SHEET**

**Name:** \_\_\_\_\_

**Company:** \_\_\_\_\_

**Hire Date:** \_\_\_\_\_

**Position:** \_\_\_\_\_

**Chemical Surety Date** \_\_\_\_\_

**Refresher Date** \_\_\_\_\_

**HWM Refresher Date** \_\_\_\_\_

**DAAMS GB Cert Date** \_\_\_\_\_

**DAAMS VX Cert Date** \_\_\_\_\_

**DAAMS Update** \_\_\_\_\_

**DAAMS H Cert Date** \_\_\_\_\_

**Brines GB Cert Date** \_\_\_\_\_

**Brines VX Cert Date** \_\_\_\_\_

**Brines Update** \_\_\_\_\_

**Brines H Cert Date** \_\_\_\_\_

**pH Cert Date** \_\_\_\_\_

**pH Update** \_\_\_\_\_

**% Chlorine Cert Date** \_\_\_\_\_

**Chlorine Update** \_\_\_\_\_

**DPE/PVC Cert Date** \_\_\_\_\_

**DPE/PVC Update** \_\_\_\_\_

**TOC Cert Date** \_\_\_\_\_

**TOC Update** \_\_\_\_\_

**Lewisite Cert Date** \_\_\_\_\_

**Lewisite Update** \_\_\_\_\_

**Ignitability Cert Date** \_\_\_\_\_

**Ignitability Update** \_\_\_\_\_

**NaOH Cert Date** \_\_\_\_\_

**NaOH Update** \_\_\_\_\_

AMSCM-OPDC Form 5043-R-E, 11 Feb 04

## **Attachment 3**

### **Appendix G**

#### **References**

## REFERENCES

1. *Site, Safety Submission Information Consolidation Plan (Current Version)*
2. ANSI/ASQC Z1.4-1993
3. NIOSH, *Special Report on VX: Evaluation of Pesticide Interference and Evaluation of Conversion Pad Service Life.*
4. DD Form 1222, *Request for Analytical Results*
5. AMSCM-OPDC Form 5025, *Custody Tag.* AMSCM-OPDC Form 2350, *Analysis Report.*
6. *Quality Plan #8, Control of Monitoring and Measurement Devices.*

## **Attachment 3**

### **Appendix H**

#### **Glossary**

## **Glossary**

### **1.0 ABBREVIATIONS**

ACAMS	Automatic Continuous Air Monitoring System
AEL	Airborne Exposure Limit
ADS	Agent Destruct System
AMC-R	Army Material Command Regulation
AODR	Ammunition Operation Deficiency Report
SEL	Allowable Stack Concentration
ASCII	American Standard Code Information Interchange
BB	Building Block
BDA	Brine Drying Area
CAR	Corrective Action Report
CASARM	Chemical Agent Standard Analytical Reference Material
CCV	Continuous Calibration Verification
CD	Chemical Demilitarization
CEA	Civilian Executive Assistant
CM	Control Module
CMA	Chemical Materials Agency
CMO	Control Module Operator
CQAT	CASARM Quality Assessment Team
CQAPCAAM	CASARMS Quality Assurance Plan for Chemical Agent Air Monitoring
RL	Certified Reporting Limit (.5 AEL) (Same as LCS)
CSM	Chemical Surety Materiel
DA	Department of the Army
DAAMS	Depot Area Air Monitoring System
DCD	Deseret Chemical Depot
DFS	Deactivation Furnace System
DHHS	Department of Health and Human Services
DOD	Department of Defense
DPE	Demilitarization Protective Ensemble
ECBC	Edgewood Chemical and Biological Center
ECL	Engineering Control Level
EMD	Environmental Management Division
FAL	Found Action Level
FC	First Challenge
FID	Flame Ionization Detector
FPD	Flame Photometric Detector
GC	Gas Chromatograph
GLD	Gross Level Detector
GPL	General Population Limit
HLA	High-Level Alarm
HL	High-Alarm Level
HPD	Hewlett Packard Dynatherm System
IAW	In Accordance With

## **Glossary (Continued)**

ID	Identification
IDLH	Immediately Dangerous to Life and Health
INACCMO	In Accuracy Monitoring Report
IOP	Internal Operating Procedure
IS	Internal Standard
LCL	Lower Control Limits
LCS	Lowest Concentration Standard (Same as RL)
LL	Low-Level Alarm
LIC	Liquid Incinerator
LOQ	Limit of Quantification
LMQAP	Laboratory and Monitoring Quality Assurance Plan
LSS	Life Support System (CAMDS BB#46)
MCP	Monitoring Concept Plan
MDB	Munitions Demilitarization Building
MDF/BIF	Munitions Demilitarization Facility/Bulk Items Facility
MPF	Metal Parts Furnace
MPL	Maximum Permissible Limit of Demilitarization Protective Ensemble
MSD	Mass Selective Detector
MTF	Material Treatment Facility
NRT	Near Real Time
P&A	Precision and Accuracy
PCT	Preconcentrator Tube
PDARS	Process Data Requisition and Recovery System
pgm	Program
PMCD	Program Manager for Chemical Demilitarization
PMCS	Program Manager for Chemical Stockpile Disposal
PMECW	Program Manager for the Elimination of Chemical Weapons
PMT	Photo Multiplier Tube
ppm	Parts Per Million
PROQUIS	Professional Quality Information Systems for Business
PSC	Personnel Support Complex
psig	Pounds Per Square Inch Gage
PSM	Project System Manager
PTP	Proficiency Test Program
QA	Quality Assurance
QAC	Quality Assurance Coordinator
QAS	Quality Assurance Specialist
QAPP	Quality Assurance Program Plan
QC	Quality Control
QL	Quality Laboratory Samples
QP	Quality Plant Sample
RDT&E	Research, Development, Testing and Evaluation

### **Glossary (continued)**

RF	Response Factor
RM	Risk Management
RRF	Relative Response Factor
RTAP	Real Time Analytical Platform
SAF	Site Analytical Facility
SBCCOM	Soldier, Biological and Chemical Command
SEL	Source Emission Limit
SMI	Storage Monitoring Inspection
SAS	Statistical Analysis Software
SOP	Standing Operating Procedure
STEL	Short Term Exposure Limit
TAL	Target Action Level
TC	Target Concentration
TEAD	Tooele Army Depot
TMDE	Test Measurement and Diagnostic Equipment
TMF	Toxic Maintenance Facility (CAMDS BB#51)
TWA	Time Weighted Average
TWG	Technical Working Group
UIFM	Uncertainty In Found Mass
UCL	Upper Control Limits
USACDRA	United States Army Chemical Demilitarization and Remediation
USACSDP	United States Army Chemical Stockpile Disposal Program
VDC	Volts Direct Current
VOL	Volume
WCL	Waste Control Limit
WPL	Worker Population Limit

## 2.0 TERMS

AVG	Average
°C	Degrees Centigrade/Celsius
F	Degrees Fahrenheit
>	Greater Than
≥	Greater Than or Equal To
<	Less Than
Z	Monitoring Level
%	Percent
±	Plus or Minus
GA	ethyl N, N-dimethylphosphoramidcyanide
GB	isopropylmethylphosphonofluoridate or Sarin Gas
HCN	hydrogen cyanide
HD	Distilled mustard; distilled bis(2-chloroethyl) sulfide
L	Lewisite, dichloro-2-chlorovinyl arsine
L/min	Liter per minute
mg/m <sup>3</sup>	milligrams per cubic meter
MINICAMS™	Miniature Chemical Agent Monitor
mL	milliliter
Mustard	H, HD, or HT
ng	nanogram
RDX	Cyclotrimethylenetrinitramine
RST	Reset
TETRYL	trinitrophenylmethylnitramine
TNT	trinitrotoluene
μL	microliter
VX	O-ethyl, S-(2-diisopropylaminoethyl) methylphosphonothiolate
Z	generic monitoring level

## 3.0 SPECIAL ABBREVIATIONS AND TERMS

### **Analyte**

The substance that one is interested in detecting and/or quantifying when one performs a chemical analysis of a sample.

“Assignable Cause”

### **Blanking**

When DAAMS tubes must be thermally desorbed (at 200° C) in the heating block for 20 minutes, using a vacuum with a minimum flow rate of 0.5 liters per minute (L/min).

### **Class I Method or Monitor**

A Class I Method or Monitor is a fully quantitative analytical method or monitor that yields a numerical estimate of the analyte concentration within a working concentration range.

Class II Method or Monitor

A qualitative or semi-quantitative method or monitor that is used only to ascertain whether or not an analyte is present at or above a specific concentration.

***Compliance Level***

A compliance level value corresponds to the probability that a single future measurement will yield a particular result, or fall within a particular interval of values.

***Confidence Limits (or Bounds)***

The confidence limit is the upper and lower bounds of an interval of values within which a certain percentage of future measurements may fall.

***Confidence (or Significance) Level***

A value that corresponds to the probability that a single future measurement will yield a particular result, or it falls within a particular interval of values.

***Control Parameter***

A parameter whose value depends on the bias or imprecision (or both) of a method and whose value is used in a procedure to produce control charts to maintain daily control of method bias or imprecision (or both).

***Found Action Level (FAL)***

The highest measured or found analyte concentration at which there is a 97.5% level of confidence that the true analyte concentration is less than the hazard level.

***Gross Level Detector (GLD)***

The GLD is an engineering control level for mustard that has been established for exiting airlocks and specified locations within chemical agent contaminated areas. The level assigned is 0.2 mg/m<sup>3</sup>. This level is consistent with the hardware and software of NRT monitor and is below the 1.67 mg/m<sup>3</sup>, which correlates to the regulatory basis for IDLH values or other chemical agents. An IDLH has not been assigned to chemical agents HD and L because of carcinogenic properties.

***Hazard Level***

The hazard level is a control limit for a hazardous substance (analyte) in an air matrix. For example, the SEL for chemical agent GB is the hazard level for this agent in stack gas.

***High-Level Alarms (HLA)***

HLA are automatic detectors that alarm for IDLH levels of chemical agents GB and VX in 130 seconds. Chemical agents HD GLD detectors alarm in 5 minutes. These detectors are used in non-toxic areas of the plant. They provide warning for chemical agent concentrations of immediate concern.

***Low-Level Alarms (LL) Systems***

LL Systems are automatic detectors that alarm to extremely low levels of chemical agent (STEL or SEL). These detectors are used throughout the plant in areas where there is potential for chemical agent contamination. They are used to monitor allowable exposure levels (TWA) to minimize exposure of personnel to these concentrations. Monitoring at these levels provides the earliest indication of process-upset conditions. These monitoring levels are operationally treated as ceiling values for the purpose of masking workers at the CAMDS Site.

***Historical Samples***

Historical samples are gathered from locations that are downstream from NRT/DAAMS monitoring stations. These historical samples are normally DAAMS only stations that have been previously monitored, such as the stations inside and outside of the toxic areas. The LSS air

reconnect stations and the Deactivation Furnace System (DFS) cyclone dunnage bin are exceptions where DAAMS only stations are not simply for historical samples. Twelve (12) hour DAAMS also are used as historical samples at the perimeter stations to sample for the presence of chemical agents at the CAMDS Site and ammunition storage areas.

#### ***Maximum Permissible Hazard Limit (MPL)***

The MPL is the highest chemical agent challenge used in testing of the demilitarization protective ensemble (DPE), used in toxic areas of demilitarization plants. Consequently, demil personnel are not allowed to enter areas in excess of the 100.0 mg/m<sup>3</sup> level for chemical agents GB, VX, and HD. A low volume sampler is used to dilute the chemical agent so that it is within calibration range of the NRT monitors.

#### ***Method Bias***

Method bias is a systematic error inherent in a method or caused by some artifact or idiosyncrasy of the measured system. Examples are temperature effects, extraction inefficiencies, contamination, mechanical losses, and calibration errors. Bias may be both positive and negative, and several kinds can exist concurrently so that net bias is all that can be evaluated, except under special conditions.

#### ***Method or Measurement Accuracy***

The method or measurement accuracy is a degree of agreement of a measured value with the true or expected value of the quantity of concern. Method accuracy depends on both the bias and the imprecision of the method.

#### ***Method or Measurement Inaccuracy***

The magnitude of the deviation of a single measurement result from the corresponding true value, or the magnitude of deviation from the true value that is expected to be exceeded by no more than (100-X)% of future measurements (where X% is the applicable confidence level). If the data are normally distributed about a mean value, then method inaccuracy, as defined in the second definition above, is approximately equal to the sum of the absolute value of the method bias plus k times the standard deviation, where k is the number of standard deviations above and below the average found concentration that will embrace X% of future measurement results. For example, If b is the method bias and 5 is the standard deviation, then with 95% confidence, the method inaccuracy is  $b + 2s$ .

#### ***Precision***

The degree of mutual agreement characteristic of independent measurements as the result of repeated application of the process under specified conditions.

#### ***Process Control Monitors***

Process Control Monitors are used to identify process changes. When an operation or process is performed in a controlled toxic area, the chemical agent level in the controlled area will stabilize when the operation is stable. After that time, changes in chemical agent levels will indicate changes in the process and possibly indicate upset conditions. The process control monitor will notify operators that corrections may be required.

#### ***QL***

A synthetic sample prepared by spiking (i.e., fortifying) a typical sampling device or an aliquot of a liquid sampling medium with the analyte. QLs are usually prepared in the LAB by personnel other than the analysts who are expected to analyze them.

### ***QP***

A QP is a QC sample that has been spiked with a solution of analyzed dilute chemical agent and exposed to the plant atmosphere. The QPs spike concentration will be +10% of 1.0 Z. The QP is used to assess the performance of both the ACAMS and DAAMS methods. The QPs found concentration will be  $\pm 25\%$  of the monitoring level (i.e., the target concentration). The DAAMS tubes are spiked with a QP standard at the LAB and then carried to the monitoring station. At the station, the tube is aspirated for the amount of time prescribed by the method and subsequently returned to the LAB for analysis.

### ***Random Error***

A random error is a component of total error that is not assignable to any specific source and the magnitude of which can be predicted only in terms of probability.

### ***Systematic Error***

The amount by which the mean of the distribution of measurements differs from the true or target valued measured. This error may be either dependent on, or invariant with respect to, analyte concentration.

### ***Target Action Level (TAL)***

The highest target or true analyte concentration that can be distinguished as lower than the hazard level 97.5% of the time when using a test, whose probability of a false-positive response is 0.025 (or 2.5%).